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Research Materials Developed Under the NBS Inorganic Materials Program

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Research Materials Developed Under the NBS Inorganic Materials Program

Edited by
F. E. Brinckman and J. B. Wachtman, Jr.

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Contents

	Page
1. Introduction	v
2. Definition of Research Materials	v
2.1. General Definition	v
2.2. Differences in Degree of Characterization Needed for Research on Properties With Varying Degrees of Structure Sensitivity	vi
2.3. Inhomogeneity	vi
2.4. Metastable Materials	vi
3. Relation to Standard Reference Materials	vi
4. Organization of the Tables	vi
4.1. Rationale	vi
4.2. Use of Tables—Key	vii
5. References	vii
Tables	
I. Gases	1
II. Liquids	7
III. Glasses	16
IV. Single Crystals	20
V. Polycrystals	41
VI. Multiphase Systems	61

Research Materials Developed Under the NBS Inorganic Materials Program

F. E. Brinckman and J. B. Wachtman, Jr.

The National Bureau of Standards develops many specialized materials in the process of carrying out research supporting its measurements, standards, and service activities. These materials include gases, liquids, glasses, single crystals, polycrystals, and various multiphase materials; their compositions (including trace elements in some cases) and physical characteristics are tailored to specific research needs, but the materials are often of use for other research purposes. Production is usually limited to immediate internal needs and samples are not generally available for distribution, but knowledge of production techniques and materials characteristics which may be helpful to other scientists is available. The present listing of research materials developed in the Inorganic Materials Division accordingly gives names of scientific staff members who may be contacted for this type of information as well as giving a brief summary of the nature, method of preparation and properties determined for each material.

Key words: Chemical properties; composition; gases; glasses; liquids; multiphase materials; physical properties; polycrystals; preparation; research.

1. Introduction

Improved materials are critically needed in many areas of technology. Attempts to provide such superior materials are frequently empirical and sometimes wasteful of time and funds [1].¹ An empirical component will probably always be present in any materials development program, but the establishment of a science of materials that increasingly affords predictable and reliable results in devising new materials for specific tasks is most desirable. Not only will this increase the efficiency of materials development programs, but also can ultimately lead to development of completely new materials or new applications. Much thought has been given to opportunities arising from recent developments in materials science [2] and to roadblocks preventing engineering achievement of scientifically feasible materials including the areas of characterization [1] and processing [3]. Advances in materials science assist materials development on the one hand, but depend upon development of materials suitable for research on the other. This interdependence of materials development and materials science is widely recognized and new programs on the properties of materials increasingly have as a major component a research material development subprogram. The experimental and theoretical competence needed to produce and characterize specimens is frequently quite different from that needed for the subsequent property study so that the decision to undertake a property study on a new material frequently means that a large investment of time and resources, perhaps comparable to the property measuring effort itself, must be made before the property study can begin. In these circumstances the availability of a few trial specimens, even if not of completely satisfactory quality for the final study, can be very valuable in permitting a test of the experimental feasibility of the contemplated measurements and perhaps in providing guidance on how closely the character of the specimen must be controlled for the final study. When trial specimens are not available, knowledge of previous preparation techniques and of the nature and quality of specimens which have

been produced successfully is usually quite valuable in planning and carrying out the sample preparation aspect of a new research program. The present listing of research materials produced in the Inorganic Materials Division is offered as a means of promoting direct scientist-to-scientist exchange of information on production and characterization of materials for which the division has some special competence. In some cases, specimens may be on hand and available on request; in other cases, facilities and staff may be available to produce additional specimens for purposes coming within the NBS mission. Provision of information, rather than specimens, is the principal goal, however, because maintenance of a stock of the numerous and extremely varied materials produced in small batches or maintenance of specialized production facilities on a standby basis is not feasible.

Information concerning nationwide sources of many research materials, especially single crystals, is available from the Oak Ridge Research Materials Information Center [4].

The present listing of materials may be useful to scientists and engineers interested in the production of a material or its general availability for purposes other than research. No claim is made concerning exhaustive knowledge of the materials listed, but the staff members involved in the production or characterization of unusual materials sometimes have special knowledge relevant to other applications in addition to research.

2. Definition of Research Materials

2.1. General Definition

A research material is here considered to be one sufficiently well-characterized to be useful for a particular type of current research. Ideally, a fully characterized material is desirable; that is, the character (chemical composition, structure, microstructure, etc.) should be uniform and held within such narrow and known limits that all the chemical and physical properties of the material are well determined. Practical considerations usually limit the characterization to

¹ Figures in brackets indicate the literature references on page 3.

the factors thought to control the property being studied and limit the accuracy of the characterization. Thus, a material useful at a particular stage of research may no longer be useful for the same type of research as measurement techniques improve and finer details of behavior are studied or as new factors are discovered to have an influence on properties at the level being studied. A valid research material can cease to have this distinction as a field of study progresses but the same material can sometimes again become an important research material as new phenomena are investigated. For example, ruby grown by the Verneuil process for bearings, wear surfaces, or jewels suddenly became an exciting research material when the ruby laser was invented.

2.2. Differences in Degree of Characterization Needed for Research on Properties with Varying Degrees of Structure Sensitivity.

The characterization needed for certain research, such as phase equilibria, is sometimes limited primarily to the major element composition and such factors as trace impurities or surface condition are relatively unimportant. This fact is reflected in the listing, for example, of a number of crystalline materials developed for phase equilibria studies and a number of glasses developed for bulk optical or elasticity studies; major component characterization was sufficient for these purposes. Some of these materials have subsequently become the object of renewed research interest in connection with other properties dependent primarily upon major component composition such as electro-optic properties, photoelasticity, and mechanical properties under pressure. Certain structure-sensitive properties, such as transport, plastic deformation, and fracture depend upon trace impurities and upon small deviations from stoichiometry. Samples made for research in these areas, even though of the same major component composition as some of those mentioned previously, require more careful processing and characterization and are accordingly listed as separate entries with an indication of their special features.

2.3. Inhomogeneity

Among both the bulk composition and trace impurity types there are cases where a deliberate degree of inhomogeneity is required. Thus, a series of glasses with the same total composition but differing degrees of phase separation forms an interesting family of research materials. An example of an even finer scale of deliberately produced inhomogeneity is a series of crystals of CaF_2 all having the same level of Gd additive but differing in the degree of association of point defects with Gd atoms. This last example illustrates another feature of some of the listed materials; in this case specimens of given bulk composition were produced by a commercial supplier to NBS specifications. The NBS contribution was the development of special treatments and measurement tech-

niques needed to produce and determine different degrees of association.

2.4. Metastable Materials

Still another category of research materials involves those which are not stable or have a very short lifetime under ordinary conditions but which occur as important components under special conditions such as high temperature vapor species, high pressure crystalline phases, or short-lived reactive intermediates. Such materials generally cannot be kept in stock but the technology of preparation and the techniques for measurement of concentration and properties during the brief lifetime of the material are important aspects of research material information. An overlapping family of research materials consists of those which are dangerous (toxic, explosive, or corrosive) and so require special handling procedures during processing, storage, or property measurement. An example of such a difficult research material is the high pressure polymorph of the detonator explosive lead azide.

3. Relation to Standard Reference Materials

A few of the research materials listed here have been developed into Standard Reference Materials and many of the Standard Reference Materials are used as research materials but the two categories should not be confused despite the fact that there is some overlap. Standard Reference Materials are kept in stock by the National Bureau of Standards and sold by the Office of Standard Reference Materials. Each Standard Reference Material is certified with respect to the aspects of chemical and/or physical properties relevant to the material's intended application. Perhaps the greatest use of these materials is for calibration of instruments and checking of measurement procedures, but the high degree of homogeneity and extensive characterization associated with Standard Reference Materials frequently makes them useful as research materials. A full listing of Standard Reference Materials is available [5].

4. Organization of the Tables

4.1. Rationale

As noted in the Introduction, entries have been tabulated into sections derived chiefly from gross physical state at ordinary conditions (e.g., gases, polycrystals, multiphases, etc.). Clearly a number of exceptions occur, particularly for research materials of low stability or transient existence, but their location will be fairly obvious to the reader.

Since this compilation tends to emphasize composition rather than properties, special care was taken in organizing the tables. Use of the widely accepted Chemical Abstracts Formula Index does not always generate familiar empirical chemical formulae, nor

does this system readily lend itself to classification of glasses, but we chose it for its rational basis. Moreover, significant progress has been achieved for computer searching and retrieval; indeed, the parent program is now in use by the Patent Office.

4.2. Use of Tables—Key

a. The arrangement of symbols in formulae is alphabetic except that in carbon compounds C always comes first followed immediately by H if hydrogen is also present.

b. The arrangement of formulae or entries is also alphabetic, except that the number of atoms of any specific kind influences the order of listing; e.g., all C_1 compounds appear before C_2 , thus CCl_3O , CCl_4 , $CHCl_3$, CHN , CH_2O , CO , C_2Ca , $C_2H_2O_2$.

c. Water of hydration is not made a part of the formulae indexed.

d. Polymers having different names and recognized as different substances, e.g., acetaldehyde and paraldehyde, are all entered under their accepted formulae; but a definite compound for which different polymeric formulae are in use is entered under the simplest formula.

e. For series of compounds listed under a single entry heading "M" refers to metallic or metalloid components (e.g., K, Li, Rb) and "X" denotes electronegative substituents such as Cl, F, O, S, etc.

f. Approximate compositions are indicated by enclosing listing in quotation marks, "—", such as in interstitial compounds or alloys.

g. Glasses, polyphases, and compounds of given stoichiometry but indefinite structure are cited alphabetically by M, followed by X; thus $B_2O_3 \cdot 3Nb_2O_5$; $Cr_2O_3 \cdot IrO_2$; $B_2O_3 \cdot BaO \cdot SiO_2$; etc.

h. Isotopic compositions are presumed to be of terrestrial abundance unless otherwise indicated.

i. Wherever practicable conventional formulae and/or nomenclature is listed to the right of the index formula.

j. Great effort has been made to maintain accuracy and provide current information. In some instances, through staff changes, some authors cited are no

longer in the Inorganic Materials Division, yet inclusion of their recent efforts was deemed significant to this compendium. Hence, these individuals are signified by an asterisk. The reader may contact these individuals for further information, but it would probably be more expedient if the editors were contacted.

Many of the materials listed in this report were produced as part of programs sponsored by other agencies. Acknowledgements for specific materials are given in the references and are too numerous to repeat in detail here. The support by the Air Force Materials Laboratory, the Air Force Cambridge Research Laboratory, the Army Research Office (Durham), the Advanced Projects Research Agency (Materials Office), the Atomic Energy Commission (Research Division), the National Aeronautics and Space Administration, and the Office of Naval Research is gratefully acknowledged.

5. References

- [1] Characterization of Materials, prepared by the Committee on Characterization of Materials of the Materials Advisory Board, H. W. Leverenz, Committee Chairman, National Academy of Sciences—National Academy of Engineering Publication MAB-229-M, March 1967.
- [2] Research in Solid-State Sciences—Opportunities and Relevance to National Needs, prepared by a Committee of the Solid State Sciences Panel for the Coordinating Committee for Materials Research and Development of the Office of Science and Technology, R. Smoluchowski, Committee Chairman, Publication 1600, National Academy of Sciences, 1968.
- [3] Ceramic Processing, prepared by the Committee on Ceramic Processing, of the Materials Advisory Board, Joseph A. Pask, Committee Chairman, Publication 1576, National Academy of Sciences, 1968.
- [4] J. W. Cleland, Purity and Perfection of Research Specimens of Oxides in Mass Transport in Oxides, Nat. Bur. Stand. (U.S.), Spec. Pub. 296, (224 pages) edited by J. B. Wachtman, Jr. and A. D. Franklin, issued August 1968. The address of the Research Materials Information Center is Building 3001, Oak Ridge National Laboratory, Box X, Oak Ridge, Tennessee 37830.
- [5] Catalog of Standard Materials, Nat. Bur. Stand. (U.S.), Spec. Publ. 260, July 1970. See also supplements for price lists and changes.

NBS STAFF MEMBERS

TO BE CONTACTED
FOR ADDITIONAL
INFORMATIONREFERENCES (Author,
Title, Journal)

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	REFERENCES (Author, Title, Journal)
$\text{BCl}_n\text{F}_{3-n}$	equilibrium soln. of all species, where $n = 0-3$, studied as liquids	$\text{BCl}_3 + \text{BF}_3 \rightleftharpoons$ at $< 25^\circ\text{C}$	NMR (^{11}B , ^{19}F) spectrometry	attempt to determine chemical exchange rates in equilibrium	(1) T. C. Farrar and T. D. Coyle, "Proton and Fluorine NMR Spectra of HBF_2 ", J. Chem. Phys., <u>41</u> , 2612 (1964).
BF_2H	also studied as liquid (1,2)	(3) $\text{BF}_3 + \text{B}_2\text{H}_6$ at 250°C (4) $\text{BF}_3 + (\text{CH}_3\text{O})_2\text{BH}$ $\text{B}_2\text{F}_4 + (\text{CH}_3)_3\text{SiH} + h\nu$	mol. wt., infrared NMR (^1H , ^{11}B , ^{19}F) spectrometry, active H, chemical reactivity	qualitative structural detms; only known monoboron fluoro-hydride; synth. reagent; air/moisture sensitive	(2) E. B. Whipple, T. H. Brown, T. C. Farrar, and T. D. Coyle, "Relative Signs of Nuclear Spin Coupling in $^1\text{H}^1\text{B}^1\text{F}_2$ ", J. Chem. Phys., <u>43</u> , 1841 (1965).
HBF_2					(3) T. D. Coyle, J. J. Ritter, and T. C. Farrar, "Preparation and Properties of Difluoroborane", Proc. Chem. Soc., <u>25</u> , (1964).
difluoroborane					(4) T. D. Coyle, J. Cooper, and J. J. Ritter, "Preparation and Some Reactions of Difluoroborane", Inorg. Chem., <u>7</u> , 1014 (1968).

BF_5OSi BF_2OBF_2	electric discharge rxn of BF_3 in SiO_2 reactor	infrared, mass spectrometry using full isotopic anal.	novel Si-B-B molecular structure suitable for bond angle and dissn. studies; unstable at r.t.	F. E. Brinckman and G. Gordon	(1) F. E. Brinckman and G. Gordon, "Energetic Intermediates in Inorganic Synthesis: Reactions of Covalent Main Group Fluorides in Electric Discharges", Fourth Int. Symp. on Fluorine Chem., Estes Park, Colo., July 1967, Abstr. of Papers, p. 132.
B_2F_4 diboron tetrafluoride tetrafluorodiborane-4	also studied as liquid $\text{B}_2\text{Cl}_4 + \text{SbF}_3$ at -80°C	infrared, mass, NMR (^{11}B , ^{19}F) spectrometry	simplest polyboron fluoride; potential source of BF_2 function in free radical reactions; B-B bond dissn. energy	T. D. Coyle T. C. Farrar	(2) F. E. Brinckman and G. Gordon, "Energetic Intermediates in Inorganic Synthesis: Characterization of Transport Species in Electric Discharges", Proc. Int. Symp. on Decomposition of Organometallic Compounds to Refractory Ceramics, Metals, and Metal Alloys, Univ. Dayton Press (1967), p. 29.
$\text{B}_2\text{F}_4\text{O}$ BF_2OBF_2	obsd. as parent-daughter ions microwave discharge of BF_3 in SiO_2 reactor	direct inlet mass spectrometry, isotopic anal.	novel B-O-B molecular structure suitable for bond angle and dissn. measurements; unstable even at low pressures	F. E. Brinckman	See refs. 1, 2 under BF_5OSi .

F_2H_6	studied as liquid	$\text{nBF}_3 \cdot \text{O}(\text{C}_2\text{H}_5)_2$ + LiAlH_4 + (n = 10 or 11)	infrared (1), NMR (^1H , ^{10}B , ^{11}B) spectrometry (2)	parent material for synthesis of selected isotope (^1H , ^{10}B , ^{11}B) boranes; de-tailed interpretation of NMR spectra	T. D. Coyle T. C. Farrar R. B. Johannesen	(1) W. J. Lafferty, A. G. Maki, and I. D. Coyle, "High Resolution Infrared Spectrum and Structure of Diborane", J. Mol. Spectroscopy, <u>33</u> , 345 (1970). (2) T. C. Farrar, R. B. Johannesen, and T. D. Coyle, "Magnetic Non-Equivalence in the High Resolution NMR Spectra of Diborane", J. Chem. Phys., <u>49</u> , 281 (1968). See also $\text{C}_2\text{H}^{11}\text{BD}_6\text{O}_2$ in SECTION II.
diborane						

CCl_3F	also studied as liquid, solid	commercial	infrared, NMR (^{19}F , ^{35}Cl , ^{37}Cl) spectrometry, volatility, m.p.	model compound for structure in liquid; anisotropic motions in liquids; angular momentum cross-sections	T. C. Farrar	
Freon-11						
CHN		$\text{NaCN} + \text{H}_2\text{SO}_4$	infrared and mass spectrometry	used for photoionization studies: parent compound for X-CN series	T. D. Coyle	V. H. Dibeler and S. K. Liston, "Mass Spectrometric Study of Photoionization. IX. Hydrogen Cyanide and Acetonitrile", J. Chem. Phys., <u>48</u> , 4765 (1968).
HCN						
CH_3NO_2		$\text{NaNO}_2 + \text{CH}_3\text{OH} + \text{H}_2\text{SO}_4$	infrared, ultraviolet, mass spectrometry; vapor phase chromatography	prepd. for photoionization study	T. D. Coyle	
CH_3ONO						
methyl nitrite						

C_2HBCl_2	ethynyldichloro- borane	$trans-ClCH = CHCl_2 + h\nu$	mass, infrared spectrometry; cleavage with propionic acid $\rightarrow C_2H_2 + HCl$	first ethy- nylboron chloride iso- lated; novel substituted acetylene; suitable for studies on coordination of saturation of B; air/moisture sensitive	J. J. Ritter T. D. Coyle	J. J. Ritter, T. C. Coyle, and J. M. Bellama, "Synthesis of Ethynylboron Halides", Chem. Comm., 908 (1969).
C_2HBF_2	ethynyldifluoro- borane	C_{2v} symmetry	$(CH_3)_3SnC \equiv CH + BF_3$ at -80°C	mass, infrared, and microwave spectrometry, cleavage in glass with propionic acid + $C_2H_2 + SiF_4$	first ethy- nylboron fluoride iso- lated; suitable for extensive structural anal.; air/ moisture sensitive	See ref. under C_2HBCl_2 . W. J. Lafferty, J. J. Ritter, "Microwave Spectrum, Structure, and Dipole Moment of Ethynyldifluoro- borane, $HC\equiv CBF_2$ ", Chem. Comm., 909 (1969).
$ClFO_3$ $FClO_3$ perchloryl fluoride		also studied as liquid, solid	commercial	see CCl_3F	T. C. Farrar	
$ClF_{n+1}Si$ chlorofluoro- silanes	$n = 1-3$ also studied as liquids	$SiCl_4 + AlCl_3$ (cat.) at 150°C (1) electric dis- charge in $SiCl_4$ wt.: vapor phase + SiF_4 (2)	infrared, mass (full isotopic anal.), NMR [^{19}F , (^{29}Si)] spectrometry; volatility, mol. wt.: vapor phase chromatography	model com- pounds for structural, dipole moment ($n = 1, 3$), and magnetic resonance properties; synthetic intermediates	F. E. Brinckman T. D. Coyle T. C. Farrar	(1) R. B. Johannessen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. V. Fluorosilanes", J. Phys. Chem., 72, 660 (1968). (2) See refs. under BF_5OSi . (3) See ref. under F_4Si .

D	D(atoms)	microwave discharge in $D_2O(g)$	measurement of electron g-factor relative to g_f of H atoms	experimental	M. Linzer	"Measurement of the Proton g-factor in Cyclohexane and the Electron g-factors in Atomic Deuterium and Atomic Nitrogen", Bull. Am. Phys. Soc., <u>12</u> , 507 (1967).
		$PF_2I + HI$ (1)	mol. wt.; infrared NMR ($^1H, ^{19}F, ^{31}P$)(2) spectrometry	NMR relative sign measurements; reagent uses; air/moisture sensitive	T. D. Coyle	(1) R. W. Rudolph and R. W. Parry, "Fluorophosphate Ligands. I. The Preparation and Characterization of Difluorophosphate", Inorg. Chem., <u>4</u> , 1339 (1965). (2) R. B. Johannesen, "NMR Studies of Inorganic Fluorides. IV. Relative Signs of Coupling Constants in CH_3SiF_3 , $HSiF_3$, and HPF_3 ", J. Chem. Phys., <u>47</u> , 3088 (1967).
	F_3OP OPF_3	studied as liquids also	infrared, mass (1), NMR ($^{19}F, ^{31}P$)(2) spectrometry; volatility	related to study of electric discharge rxns in PF_3 (1); model compounds for study of structures and electron anisotropies (2)	F. E. Brinckman T. C. Farrar (2)	(1) F. E. Brinckman, and G. Gordon, "Formation of μ -Oxyfluorophosphines and Polyphosphines in Gaseous Discharge Reactions", 4th Middle Atlantic Regional Mtg of the Am. Chem. Soc., Wash. D. C., February 1969, Abstr. of Papers, p. 41.
	F_3P PF_3	Also obsd. as liquid (1)	infrared, mass, NMR (^{19}F) spectrometry; volatility, mol. wt., vapor phase chromatography	Si-F coupling constant; 29Si NMR by double resonance	T. D. Coyle T. C. Farrar	T. D. Coyle, R. B. Johannesen, F. E. Brinckman, and T. C. Farrar, "NMR Studies of Inorganic Fluorides. II. Solvent Effects on J(29Si-19F) in SiF_4 ", J. Phys. Chem., <u>70</u> , 1682 (1966).
	SiF_4 tetrafluorosilane					

F_3GeO_2 (GeF_3) ₂ O	obsd as parent and daughter ions	microwave dis- charge rxns. of GeF_4 and $GeF_4 + SiF_4$ in SiO_2 reactors	direct inlet mass spectrometry, full isotopic anal.	candidate for molecular structure and bond dissn. energy measure- ments; yields very small	F. E. Brinckman	See ref. 2 under BF ₃ OSi.
F_6GeOSi GeF ₃ OSiF ₃						
F_6OSi_2	also studied as liquid	$Si_2OCl_6 +$ $SbF_3(l)$; electric discharge rxn of SiF_4 with $SiO_2(2)$	NMR [¹⁹ F, (¹⁹ F(²⁹ Si))] (1); mass (2) spectrometry	suitable for determination of Si-O-Si bond angle; purified by vapor phase chromatography; air/moisture sensitive	F. E. Brinckman	(1) R. B. Johannesen, T. C. Farrar, F. E. Brinckman, and T. D. Coyle, "NMR Studies of Inorganic Fluorides. I. High-Resolution ¹⁹ F Spectra of Si_2F_6 and (SiF_3) ₂ O", J. Chem. Phys., <u>44</u> , 962 (1966). (2) See ref. 2 under BF ₃ OSi.
Si_2OF_6 hexafluoro- disiloxane						
F_6Si_2	also studied as liquid	$Si_2OCl_6 +$ $SbF_3(l)$	NMR [¹⁹ F, (¹⁹ F(²⁹ Si))] (1); spectrometry chemical reactivity (2)	parent com- pound for perfluoro- disilanyl chemistry; suit- able for detm. of Si-Si bond energy; purified by vapor phase chromatography; air/moisture sensitive	F. E. Brinckman	(1) See ref. 1 under F ₆ OSi ₂ . (2) F. E. Brinckman, T. D. Coyle, and L. Fishman, "Forma- tion of a Perfluoro- disilanyl-silazane: Selected Cleavage Reactions of Silazanes", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Abstr. of Papers, p. D14.
Si_2F_6 hexafluorodisilane						
Ga_2O		$Ga_2O_3(s)$ heated to 1200-1300°C	mass spectrometry		R. C. Paule	
Ga_2O_W Ga_2WO_4		$Ga_2O_3(s) +$ W heated to 1200°C	mass spectrometry		R. C. Paule	
N	N(atoms)	microwave discharge in N ₂ (g)	see D	see D	M. Linzer	See ref. under D.

II. LIQUIDS

NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION				REFERENCES (Author, Title, Journal)	
COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	
$\text{BF}_3 \cdot \text{F}_3\text{H}_3\text{P}$ $\text{F}_3\text{P} \cdot \text{BH}_3\text{D}_n\text{F}_3\text{P}$	$n = 1-3$ (2)	$\text{PF}_3 \cdot \text{B}_2\text{H}_6$ (deuterated)	synthesis, microwave spectrometry	rotational spectrum, barrier to internal rotation	R. Kuczkowski and D. Lide, "Microwave Spectrum, Structure, Dipole Moment, and Barrier to Internal Rotation of Phosphorus Trifluoride-Borane", J. Chem. Phys., <u>46</u> , 357 (1967).
BF_3Si_2 $\text{SiF}_3\text{SiF}_2\text{BF}_2$		prepd. at Rice Univ. (a) $\text{SiF}_2(\text{g}) +$ $\text{BF}_3(\text{g})$ (b) co- condensed at -196°C	infrared, NMR (^{11}B , ^{19}F) spectro- metry	series of novel Si-B compounds; qualitative detm. of structure; air/moisture sensitive	F. E. Brinckman P. L. Timms, T. C. Ehler, J. A. Margrave, F. E. Brinckman, F. C. Farrar, and T. D. Coyle, "Silicon- Fluorine Chemistry. II. Silicon-Borane Fluorides, J. Am. Chem. Soc., <u>87</u> , 3819 (1965).
BF_9Si_3 $n\text{-Si}_3\text{F}_7\text{BF}_2$					

B_2Cl_4	unstable liquid	electric discharge reduction of BCl_3 (1)	infrared, mass (2) spectrometry; chemical reactivity (3)	parent material for chemistry of diboron compounds; detm. of B-B bond energy	J. J. Ritter	(1) T. Wartik, et al., "Diboron Tetrachloride", Inorg. Syn. 10, 118 (1967); (2) V. H. Dibeler and J. A. Walker, "Mass Spectrometric Study of Photoionization. XIII. Boron Trichloride and Diboron Tetrachloride", Inorg. Chem., 8, 50 (1969); (3) T. D. Coyle and J. J. Ritter, "Reactions of Diboron Tetrahalides with Some Unsaturated Organometallic Compounds", J. Organomet. Chem., 12, 269 (1968).
tetrachlorodiborane-4						

BrF_3-nSi	$n = 1-3$ studied as liquids and gases	$SiF_4 + SiBr_4 + AlBr_3$ catalyst at $150^\circ C$	infrared, mass spectrometry; NMR [^{19}F , (^{29}Si)] (1,2) spectrometry; volatility	dipole moments defmd. for SiF_3Br and $SiBr_3F$ (with A. Marryot, NBS)	F. E. Brinckman T. D. Coyle	(1) T. D. Coyle, F. B. Johannesen, F. E. Brinckman, and T. C. Farrar, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. II. Solvent Effects on $J(^{29}Si-^{19}F)$ in Silicon Tetrafluoride", J. Phys. Chem., 70, 1682 (1966). (2) R. B. Johannesen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. V. Fluorosilanes", J. Phys. Chem., 72, 660 (1968).
bromofluorosilanes						

$\text{CH}_3\text{B}_2\text{Cl}_3$	unstable	$\text{B}_2\text{Cl}_4 + (\text{CH}_3)_4\text{M}$ (M = Ge, Sn, or Pb)	infrared, mass spectrometry; cleavage rxns. with $\text{Ag}_2\text{O} + \text{NH}_3(\text{aq}) + \text{CH}_4 + \text{H}_2$	unsymmetrical diboron halide; useful for studies of additions to olefinic and acetylenic systems; air/moisture sensitive	J. J. Ritter T. D. Coyle	T. D. Coyle and J. J. Ritter, "Recent Studies in Organoboron Chemistry. Some Chemical Consequences of the Boron Subhalides", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Proc. of the Conf., p. R1.
$^{13}\text{CH}_3\text{I}$ $^{13}\text{CH}_3\text{O}^{13}\text{CH}_3\text{OH}$ [^{13}C]-methanol	also studied as gas	commercial, high-vacuum degassed	infrared, NMR (^1H , ^{13}C) spectrometry; m. p.	model compounds for study of ^{13}C T ₁ and T ₂ values	T. C. Farrar	See also $\text{C}_2\text{H}_4\text{O}_2$.
$\text{C}_2\text{H}^+\text{BD}_6\text{O}_2$ (CD_3O) ₂ ⁿ BH	n = 10, 11; also studied as solid	$\text{CD}_3\text{OH} + \text{nB}_2\text{H}_6$	infrared, NMR (^1H , ^{11}B) spectrometry; mol. wt.; m. p.	isotopic composition provides selected nuclear spin properties only example of B-H bond distance measurement in 3-coordinate boron compounds; motional studies; precursors of HBF_2	T. D. Coyle T. C. Farrar	(1) T. C. Farrar, J. Cooper, and T. D. Coyle, "Proton Broad-Line NMR Study of [$^{12}\text{H}_6$]Dimethoxy- ^{11}B borane", Chem. Comm., 610 (1966). (2) N. Boden, H. S. Gutowsky, J. R. Hansen, and T. C. Farrar, "Nuclear Magnetic Relaxation Studies of (CD_3O) ₂ BH", J. Chem. Phys., <u>46</u> , 2849 (1967). (3) T. C. Farrar and T. Tsang, "A Nuclear Magnetic Resonance and Relaxation Study of Dimethoxyborane", J. Res. NBS, <u>73A</u> , 195 (1969).

$\text{cis-}(\text{BCl}_2)_2 \cdot \text{H}_2 + h\nu$ $\text{trans-}(\text{BCl}_2)_2\text{CH=CHCl}$	<p>infrared, mass, NMR spectrometry; cleavage rxn of deuterio-compound with $\text{Ag}_2\text{O} + \text{NH}_3(\text{aq})$ + trans-dideuteroethylene</p>	<p>established the mode of addition of B_2Cl_4 to C_2H_2; pointed up the utility of ^1H NMR while irradiating at the boron frequency in distinguishing between 1,1 and 1,2 isomers while demonstrating the dangers of assigning structures on the basis of JH-H; demonstrated utility of $\text{Ag}_2\text{O}/\text{NH}_3(\text{aq})$ as B-C cleaving agent with retention of configuration; air/moisture sensitive</p>	<p>J. J. Ritter, T. D. Coyle</p>	<p>T. D. Coyle and J. J. Ritter, "Structure, Isomerization, and Cleavage of 1,2-Bis-(dichloroboryl)-ethylene", J. Am. Chem. Soc., <u>89</u>, 5739 (1967).</p>
$\text{C}_2\text{H}_2\text{B}_2\text{Cl}_4$ 1,1,2,2-tetrakis (dichloroboryl) ethane	<p>white solid, melts at 30°C</p> <p>$\text{ClCH=CHBCl}_2 + 2\text{B}_2\text{Cl}_4$ or $\text{HC}\equiv\text{CH} + 2\text{B}_2\text{Cl}_4$</p>	<p>elemental anal. mol. wt., rxn. ratios</p> <p>the reaction between B_2Cl_4 and halo-olefins to produce a known compound; air/moisture sensitive</p>	<p>J. J. Ritter, T. D. Coyle</p>	<p>(1) J. J. Ritter, T. D. Coyle, J. M. Bellama, "The Inter-action of B_2Cl_4 with Halo-olefins", Abstracts, 156th National Meeting of Amer. Chem. Soc., Atlantic City, N. J., (1968) p. INOR 166;</p> <p>(2) C. Chambers, A. K. Holliday, and S. M. Walker, "1,2 Tetrakis(dichloroboryl)-ethane", Proc. Chem. Soc., 286 (1964).</p>

$C_2H_3B_2Cl_2MX_3$ (M=C, Si, Ge, Sn; X=Cl, CH ₃)	involatile liq. or oils	$B_2Cl_4 + CH_2 =$ CHMX ₃	elemental anal.; mol. wt., rxn. ratios	provides series of model com- pounds for structure- reactivity relationships in vicinal- and geminal- substituted organo- metal systems	J. J. Ritter T. D. Coyle	See ref. (3) under B ₂ Cl ₄ .
$C_2H_3B_3Cl_6$ 1,1,2-tris(dichloro- boryl)-ethane		$CH_2 = CHCl +$ $2B_2Cl_4$ or $CH_2 = CHBCl_2$ $+ B_2Cl_4$	complete elemental anal.; mol. wt.; infrared, NMR spectrometry, rxn. ratio	demonstrated a reaction pathway be- tween B ₂ Cl ₄ and halo olefins contrary to earlier reports of no reaction; suggested "BCl" as a possible "reactive inter- mediate" respon- sible for the observed product; air/moisture sensitive	J. J. Ritter T. D. Coyle	(1) See ref. under $C_2H_3B_2Cl_2MX_3$. (2) See ref. (1) under $C_2H_2B_4Cl_4$.
$C_2H_4O_2$ CH ₃ ¹³ COOH a[¹³ C]-acetic acid		commercial; high-vacuum degassed	infrared, NMR (H, ¹³ C) spectro- metry; m.p.	model com- pound for study of ¹³ C I ₁ and I ₂ values	T. C. Farrar	See also ¹³ CH ₃ I and ¹³ CH ₄ O.

$C_4H_{10}F_3NSi_2$ $(CH_3)_3SiHNSiF_3$	$[(CH_3)_3Si]_2NH$ + SiF_4	infrared, mass, NMR ($^1H, ^{19}F$) spectrometry, elemental anal., sub- sequent rxns.	F. E. Brinckman See ref. 2 under BrF_3-n .	model com- pound for preparative route to perfluorosilazanes
$C_3H_{10}F_3NSi_3$ $(CH_3)_3SiHNSi_2F_5$	$[(CH_3)_3Si]_2NH$ + Si_2F_6		F. E. Brinckman, T. D. Coyle, and L. Fishman, "Formation of a Perfluoro- disilanyl-silazane: Selected Cleavage Reactions of Sila- zanes", Fourth Int. Conf. on Organo- metallic Chemistry, Bristol, England, July 1969, Proc. of the Conf., p. D14.	model compound for derivative chemistry of perfluoropolysilanes based cleavage of Si-N
C_4HCOO_4 $HCo(CO)_4$	studied as solid; liquid unstable at > $-45^\circ C$ see ref. (1)	NMR (1H) spectrometry (1,2)	(1) T. C. Farrar, F. E. Brinckman, D. Coyle, A. Davidson and J. W. Faller, "A Broad-Line Proton Magnetic Resonance Study of Cobalt Tetra- carbonyl Hydride", Inorg. Chem., <u>6</u> , 161 (1967). (2) D. L. VanderHart, H. S. Gutowsky, and T. C. Farrar, "Dipole- Dipole Interactions of a Spin 1/2 Nucleus with a Quadrupole- Coupled Nucleus", J. Am. Chem. Soc., <u>89</u> , 5056 (1967).	model com- pound for application of NMR in detm. metal- H bond dis- tances; basis for reexamina- tion of basic theory; see $C_5H_5MnO_5$
$C_4H_2FeO_4$ $H_2Fe(CO)_4$	studied as liquid	infrared, NMR (1H) spectro- metry	F. E. Brinckman T. C. Farrar	candidate compound for Fe-H distance detm., J_{Fe-H}

$C_6H_9B_3$ C,3-dimethyl-1,2-dicarbaclovo-pentaborane(5)	also studied as solid	supplied by Prof. R. N. Grimes, Univ. of Virginia, Charlottesville, Va.	NMR (1H , ^{11}B) spectrometry	clarification of molecular structure	T. C. Farrar	R. N. Grimes, "A New Isomer of $C_2B_4H_5C$, 3-Dimethyl-1,2-dicarbaclovo-pentaborane(5)", J. Am. Chem. Soc., 88, 1070 (1966).
$C_5H_4MnO_5$ $HfMn(CO)_5$		prepared at Mass. Inst. Tech., and at NBS by F. E. Brinckman	infrared, NMR (1H) spectrometry	model compound for application of NMR in detm. metal-H bond distance; see also C_4HCoO_4	T. C. Farrar	T. C. Farrar, Sister W. Ryan, A. Davidson, and J. W. Fallor, "Manganese-Hydrogen Bond Distance in $HfMn(CO)_4$ ", J. Am. Chem. Soc., 88, 184 (1966).
C_5HReO_5 $HRe(CO)_5$						
$C_6H_5N_3Si$	n = 3	(a) CH_3N_3 + CH_3MgCl (b) a + $(CH_3)_nSiCl_{4-n}$ at -40 to -80°C; see ref. (1)	infrared, NMR (1H) spectrometry (1,2), elemental anal., mol. wt.	parent compounds for synthesis of 1,3-dimethyl-1,3-dimethyl-triazeno-metal(loid) deriv.; novel bidentate ligand for coordination studies; air/moisture sensitive	F. E. Brinckman	(1) F. E. Brinckman, H. S. Haiss, and R. A. Robb, "Metal-Nitrogen Bonding, Covalent Complexes of 1,3-Dimethyltriazene with Elements of Groups I, II, III, IV, and V", Inorg. Chem., 4, 936 (1965). (2) F. E. Brinckman, K. Nelson, R. Barefoot, and H. Haiss, "Silicon-Nitrogen Bonding. Penta- and Hexa-coordinated Intermediates in Methylsilazanes", in preparation.
$C_6H_8N_6Si$	n = 2					
$(CH_3)_nSi(CH_3N=NCH_3)_{4-n}$						

C ₆ H ₆ benzene	studied also as solid	commercial, spectro-grade	NMR (¹ H, ¹³ C) spectrometry	applicat ^{ion} of Fast-Fourier transform NMR spectrometry to natural abundance ¹³ C	T. C. Farrar	See also C ₆ H ₆ under SECTION IV.
C ₆ H ₁₂ cyclohexane			NMR (¹ H) spectrometry	measurement of ¹ H g-factor relative to g _J of H atoms; secondary standard for g-factor detms; check on calculation of shielding parameter for H ₂ molecule	M. Linzer	M. Linzer, "Measurement of the Proton g-factor in Cyclohexane and the Electron g-factors in Atomic Deuterium and Atomic Nitrogen", <u>Bull. Am. Phys. Soc.</u> , <u>12</u> , 507 (1967).
Cl ₄ V VCl ₄	obsd. as neat liquid and in TiCl ₄ soln.; also obsd. as polycrystalline solid	commercial, fractionally distilled	EPR spectrometry (as a function of frequency, temp.); magnetic susceptibility between 78-307°K; optical spectrophotometry of VCl ₄ in TiCl ₄	detm. magnetic parameters, Jahn-Teller distortion; paramagnetic relaxation time, T ₁ ; air/moisture sensitive	R. B. Johannessen	R. B. Johannessen, G. A. Candela, and T. Tsang, "Jahn-Teller Distortion: Magnetic Studies of Vanadium Tetra-chloride", <u>J. Chem. Phys.</u> , <u>48</u> , 5544 (1968).
Cl ₅ FSi ₂ Si ₂ Cl ₅ F pentachlorofluorodisilane		Si ₂ Cl ₆ + SiF ₄ + AlCl ₃ (cat.) at 150°C	infrared, mass, NMR [¹⁹ F, (¹⁹ F{ ²⁹ Si})] spectrometry	first ex-ample of mixed per-halogenated disilane; ¹⁹ F "tag" permits double-resonance NMR of Si nuclei	F. E. Brinckman T. D. Coyle	See ref. 2 under BrF ₃ -Si. R. B. Johannessen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Some Fluorinated Mono- and Polysilanes", <u>153rd Natl. Mtg. Am. Chem. Soc., Miami Beach, Fla., April 1967, Abstr. of Papers</u> , p. L113.

Cl ₆ Si ₂ hexachloro- disilane	commercial, fractional distillation; electric dis- charge in SiCl ₄	infrared, mass (full isotopic anal.), spectro- metry; vapor phase chromato- graphy; volati- lity, cleavage rxn with H ₂ O → H ₂ +Cl-	parent mat- erial for synthesis of compounds bearing Si-Si bond; suitable for Si-Si bond energy detm. air/moisture sensitive	F. E. Brinckman Cooper, and T. D. Coyle, "Interactions of Some Halosilanes with Nitrogen Bases", 153rd Natl. Mtg. Am. Chem. Soc., Miami Beach, Fla., April 1967, Abstr. of Papers, p. L112.
				See SECTION I under F ₆ Si ₂ .

H ₂ O "polywater"	polymeric	not available	infrared, NMR (1H) spectrometry	attempt to establish nature of protons in sample (work with M. Malmberg, NBS)	T. C. Farvar E. D. Lippincott
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III. GLASSES

NBS STAFF MEMBERS
TO BE CONTACTED
FOR ADDITIONAL
INFORMATION

REFERENCES (Author,
Title, Journal)

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	REFERENCES (Author, Title, Journal)
barium aluminoboro-fluorosilicate glasses; example: Al_2O_3 , 12 mol%, B_2O_3 , 16 mol%, BaF_2 , 20 mol%, BaO , 8 mol%, SiO_2 , 44 mol%	microspheres, 5-50 μ dia.	melting, chilling, size reduction, flame spherizing	refractive index, thermal expansion, sphere size	filler material for dental composites	R. L. Bowen and G. W. Cleek, "X-Ray Opaque Reinforcing Fillers for Composite Materials", J. Dental Res., 48, 79 (1969).
glasses from Al_2O_3 - BaO - SiO_2 system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek
aluminosilicate glass, alkali-free	NBS Standard Reference Material # 715	melting, chilling	Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano ASTM draft in preparation.
glasses from B_2O_3 - BaO - SiO_2 system	glass blocks	melting, annealing	chemical analysis; refractive index; density; liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek E. H. Hamilton, G. W. Cleek, and O. H. Grauer, "Some Properties of Glasses in the System Barium Oxide-Boric Oxide-Silica", Amer. Ceram. Soc., 41, 209 (1958).
glasses from B_2O_3 - Ln_2O_3 system	small fragments	gravity separated from two immiscible liquids	polarizing microscopy	high-index solid state	E. M. Levin, "Liquid Immiscibility in the Rare Earth Oxide-Boric Oxide Systems", Phys. & Chem. Glasses, 7, 90 (1966).

glasses from B_2O_3 - Na_2O - SiO_2 system	melting, chilling, glass blocks	domain development kinetics; critical immiscibility temp.	specimens used for theoretical studies of immiscibility	W. Haller Blackburn, F. E. Wagstaff, and R. J. Charles, "The Metastable Immiscibility Surface in the System Na_2O - B_2O_3 - SiO_2 ", J. Am. Ceram. Soc. 53, 34 (1970).
^{10}B -enriched borate glasses doped with Co, Dy, or In	melting, chilling, grinding, glass prisms	neutron activation anal.	tested as possible method to detm. neutron flux	D. H. Blackburn "Glass Beads for Neutron Flux Measurements", NBS Technical Highlights, 1965, p. 86
glasses from BaO - La_2O_3 - SiO_2 system	melting, annealing, glass blocks	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek
a base glass: BaO 5 mol% Na_2O 15 SiO_2 75 ZnO 5 doped with various rare earths: Eu, Er, Nd	melting, chilling, annealing, blocks $3 \times 3 \times 3$ cm.	evaluation for laser use	specimens are being evaluated for fluorescence lifetime	E. J. Sharp, M. J. Weber, and G. W. Cleek, "Energy Transfer and Fluorescence Quenching in Eu and Nd Doped Silicate Glasses", J. Appl. Phys. 41, 364 (1970).
glasses from BaO - Nb_2O_5 - SiO_2 system	melting, annealing, glass blocks	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek
glasses from BaO - SiO_2 - TiO_2 system	melting, annealing, glass blocks	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek

glasses from BaO-SiO ₂ -ZnO system	glass blocks	melting, annealing	refractive index, transmittance, liquidity, temp.	specimens may be used for detm. of other properties	G. W. Cleek
alkali-line- silica glass: CaO 11.6 mol% K ₂ O 7.7 Na ₂ O 8.7 SiO ₂ 70.5	NBS Standard Reference Material # 710	melting, chilling	viscosity by beam bending	not NBS certified for this method but suitable as standard	A. Napolitano
			surface tension at elevated temp.	not NBS certified for this method but suitable as standard	A. Napolitano
			Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano
			viscosity by penetrometer	not NBS certified for this method but suitable as standard for ASTM flow point detm.	A. Napolitano
Fe-containing silicate glasses	30 x 30 x 2 mm	melting, chilling	Mossbauer spectrometry	specimens covering a wide range of Fe content may be used for characterization by Mossbauer spectroscopy	G. W. Cleek
					(1) ASTM C598 and H. E. Hagy, "Calibration of Beam Bending Ap- paratus", J. Amer. Ceram. Soc., 46, 93 (1963). (2) D. R. Secrist, "Measurement of Sur- face Tension of Glasses by a Capillary Flow Technique", Bull. Amer. Ceram. Soc., 48, 563 (1969). (3) ASTM draft in preparation. (4) J. E. Kelley, T. D. Robert, and H. M. Hanes, "A Penetro- meter for Measuring the Absolute Viscosity of Glass", U. S. Bureau of Mines RI 6358 (1964).

alkali-lead-silica glass: K ₂ O 2.6 mol% Na ₂ O 2.8 PbO 45.3 SiO ₂ 46.0	NBS Standard Reference Material # 711	melting, chilling	surface tension at elevated temp.	not NBS certified for this method but suitable as standard	A. Napolitano	See ref. 2 under alkali-lime-silica glass.
			Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano	ASTM draft in preparation.
glasses from Na ₂ O-SiO ₂ -TiO ₂ system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other glass properties	G. W. Cleek	E. H. Hamilton and G. W. Cleek, "Properties of Sodium Titanium Silicate Glasses", J. Res. NBS, <u>61</u> , 89 (1958).
porous silica glass SiO ₂	powders, 50-200 mesh particles	melting, chilling, thermal domain development, differential dissolution	mercury intrusion porosimetry, nitrogen absorption	possible surface area and porosity standards	W. Haller	(1) W. Haller, "Rearrangement Kinetics of the Liquid-Liquid Immiscible Microphases in Alkali-Borosilicate Melts", J. Chem. Phys., <u>42</u> , 686, (1965). (2) N. M. Winslow and J. J. Shapiro, "An Instrument for the Measurement of Fore-Size Distribution by Mercury Penetration", ASTM Bull., <u>39</u> (Feb. 1959).
various	glass prisms	melting, chilling, annealing, polishing	refractive index	prisms covering range of refractive indices 1.5 to 1.9 measured by minimum deviation method for N _C , N _D , and N _F	G. W. Cleek	

IV. SINGLE CRYSTALS

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
Al ₂ O ₃ alumina	also studied as powder, see SECTION V	(1) commercial powder and crystals (2) purified powder by solar furnace	melting point	melting point detm. for SRM	S. J. Schneider	S. J. Schneider and C. L. McDaniel, "Effect of Environment upon the Melting Point of Al ₂ O ₃ ", J. Res. NBS, <u>71A</u> , 317 (1967).
					S. J. Schneider	S. J. Schneider, "Cooperative Determination of the Melting Point of Al ₂ O ₃ ", Pure and Applied Chemistry, to be published.
	single crystal bar	commercial	compressibility to 10Kb	see also under BH ₃ O ₃ , SECTION V	C. E. Weir*	C. E. Weir, "Compressibility of Eleven Inorganic Materials", J. Res. NBS, <u>69A</u> , 29 (1965).
	single crystals and bi-crystals, 3 x 8 x 20 mm	chemical vapor deposition, AlCl ₃ + [O] + Al ₂ O ₃ at 1700-1750°C	spectrochemical and activation anal., optical microscopy, x-ray diffractometry	material of high physical perfection and chemical purity; suitable for diffusion studies, physical properties	H. S. Parker	H. S. Parker and C. A. Harding, "Vapor Growth of Aluminum Oxide Bicrystals", Abstracts, ACCG-NBS Conference on Crystal Growth, Aug. 11-13, 1969, Washington, D. C., p. 42.
Pure and with following dopants: Cr, Mg, Mg+Cr, Ti+Cr, Ti, V, Mn, Mn+Cr, Zn +Cr, Fe+Cr	~ 1cm	flame fusion, prepd. by D. E. Roberts	optical, EPR spectrometry	laser properties (optical and x-ray damage)	R. F. Blunt T. Chang	C. M. Stickley, et al, "Color Centers and Ruby-Laser Output-Energy Degradation", J. Appl. Phys., <u>40</u> , 1792 (1969).

$Al_{1-n}Cr_nKOs_2 \cdot 12H_2O$ $n = 1$ to 0.02 $KAl_{1-n}Cr_n(SO_4)_2 \cdot 12H_2O$	grown from aq. soln.	optical, magnetic	ferro- electric, see also $CrH_4NO_8S_2 \cdot 12H_2O$	G. A. Candela S. E. Stokowski*
AsGa GaAs gallium arsenide	highly doped and compensated epitaxy on GaAs substrate, $GaCl_3$ + $AsCl_3$ +; prepared by Monsanto Co.	spectral examination of temp. dependence of radiative recombination induced by electrical injection across p-n junction or by optical excitation	understanding of band structure of GaAs responsible for unusual absorption- edge exhibited; GaAs semi- conductor injection lasers, im- provement of room temp. performance; present efficient use of GaAs as a laser necessitates cryogenic condi- tions, cause of large decrease in efficiency at room temp. not fully understood, such studies should lead to specific material useful for improved laser performance	(1) N. N. Winogradoff and H. Kessler, "White Emission and Electrical Characteristics of Epitaxial GaAs Lasers and Tunnel Diodes", Solid State Comm., 2, 119 (1964). (2) N. N. Winogradoff, K. Owen and R. Cutnutt "The Radiative Band Pinch Effect and Temperature Dependence of Radiative Recombi- nation in GaAs", Int. J. Electronics, 22, 229 (1967). (3) N. Winogradoff, "Field Control of the Quantum Efficiency of Radiative Recombi- nation in Semicon- ductors", Phys. Rev., 138A, 1562 (1965). (4) N. Winogradoff and A. H. Neill, "Band Tailing Effects and the Temperature De- pendence of Radiative Recombination in Compensated Epitaxial GaAs Laser Junctions", IEEE J. Quant. Electronics, 4, 111 (1968).

Ba_2BaU_4 small, of tenths of mm $\text{BaO} \cdot 6 \cdot 0$	crystallized from melt of stoichiometric composition on hot wire loop	complete single crystal x-ray structure anal.	high-temp form, meta- stable at room temp.	A. Mitchell A. Perloff A. D. Mignani, A. Perloff and S. Block, "The Crystal Structure of the High Temper- ature Form of Barium Borate, $\text{BaO} \cdot \text{B}_2\text{O}_3$ ", Acta Cryst., 20, 819 (1966).
Ba_2BiO_4 $2\text{Bi}_2\text{O}_3 \cdot \text{B}_2\text{O}_3$	crystallized from melt of stoichiometric composition	complete single crystal x-ray structure anal.		A. Perloff, paper in prepn. for submission to Acta Cryst.
$\text{Ba}_2\text{O}_7\text{Pb}$ $\text{PbO} \cdot 2\text{B}_2\text{O}_3$	crystallized from melt of stoichiometric composition	unit cell and diffraction symbol from single crystal data	structure by analogy to isomorphous $\text{SrO} \cdot 2\text{B}_2\text{O}_3$	A. Perloff and S. Block, "The Crystal Structure of the Strontium and Lead Tetraborates, $\text{SrO} \cdot$ $2\text{B}_2\text{O}_3$ and $\text{PbO} \cdot$ $2\text{B}_2\text{O}_3$ ", Acta Cryst., 20, 274 (1966).
$\text{Ba}_2\text{O}_7\text{Sr}$ $\text{SrO} \cdot 2\text{B}_2\text{O}_3$	crystallized from melt of stoichiometric composition	complete single crystal x-ray structure anal.	see $\text{Ba}_2\text{O}_7\text{Pb}$	See ref. under $\text{Ba}_2\text{O}_7\text{Pb}$.
$4\text{B}_2\text{O}_3 \cdot \text{BaO}$ $\text{BaB}_6\text{O}_{13}$	melt solidi- fication, required seeding for crystallization	spectrochemical anal.; optical, x-ray powder diffractometry (from ambient to 850°C); differ- ential thermal anal.	piezo- electric at ambient temp. (ortho- rhombic form); reversibly to tetragonal form at 700°C	C. R. Robbins and E. M. Levin, "Phase Transformation in Barium Tetraborate", J. Res. NBS, 73A, 615 (1969).
$4\text{B}_2\text{O}_3 \cdot \text{Na}_2\text{O}$ $\text{Na}_2\text{O} \cdot 4\text{B}_2\text{O}_3$	crystallized from melt of stoichiometric composition on hot wire loop	complete single crystal x-ray structure anal.	high temp. form, meta- stable at room temp.	A. Hyman, A. Perloff, F. Mauer, and S. Block, "The Crystal Structure of Sodium Tetraborate", Acta Cryst., 22, 815 (1967).

BaGe ₄ O ₉	1 x 1 x x 3 mm	melt solidi- fication of stoichiometric composition	optical and x-ray diffractometry (1), including single crystal structure detm. (2)	type structure for AB ₄ O ₉ ger- manates, see also Ge ₄ O ₉ Pb and Ge ₄ O ₉ Sr in SECTION V	C. R. Robbins	(1) C. R. Robbins and E. M. Levin, "Tetra- germanates of Strontium, Lead and Barium of Formula Type AB ₄ O ₉ ", J. Res. NBS, <u>65A</u> , 127 (1961). (2) C. Robbins, A. Perloff, and S. Block, "Crystal Structure of BaGe [Ge ₃ O ₉] and its Relation to Benitoite", J. Res. NBS, <u>70A</u> , 385 (1966).
Ba ₂ Ge ₂ O ₈ Ti Ba ₂ TiGe ₂ O ₈	1 x 1 x 3 mm	melt solidi- fication and melt solidi- fication using TiO ₂ as flux (1)	spectrochemical; optical and x-ray powder diffracto- metry; differential thermal anal.	crystals exhibit pronounced domain struc- ture, show very strong piezoelectric response (1); phosphor (2)	C. R. Robbins	(1) Work in progress with particular in- terest in the crystal chemistry, domain structure and piezo- electric property of the compound. (C. R. Robbins, 1969). (2) G. Blasse, "Fluo- rescence of Compounds with Resnoite (Ba ₂ TiSi ₂ O ₈) Structure", J. Inorg. Nucl. Chem., <u>30</u> , 2283 (1968).
Ba ₂ O ₈ Si ₂ Ti Ba ₂ TiSi ₂ O ₈	1 x 1 x 5 mm	melt solidi- fication using TiO ₂ as flux (1)	spectrochemical; optical, x-ray powder diffracto- metry (powder and single crystal), differential thermal anal. (1)	piezoelectric (1); phosphor (2); type structure	C. R. Robbins	(1) C. R. Robbins, "Growth of Resnoite (Ba ₂ TiSi ₂ O ₈) From a TiO ₂ Flux and Relation to the System BaTiO ₃ - SiO ₂ ", J. Res. NES, <u>74A</u> , 229 (1970). (2) See ref. under Ba ₂ Ge ₂ O ₈ Ti.

Be ₂ CuO ₁₀ Si ₂ Y ₂ CuY ₂ Be ₂ Si ₂ O ₁₀ copper yttrium gadolinite	light blue, 1 mm; also studied as poly- crystalline material	slow cooling from Na ₂ WO ₄ or Na ₂ WO ₃ flux	precession and x-ray powder diffraction anal.	see under Be ₂ Ln ₂ MO ₁₀ Si ₂ (M = Mg, Ni, Zn, Co, Cu, Fe, Mn, Cd), SECTION V	H. S. Peiser	J. Ito, "The Synthesis of Gado- linite", Proc. Jap. Acad., <u>41</u> , 404 (1965). J. Ito, "A Note on the Gadolinite Syn- thesis", Proc. Jap. Acad., <u>42</u> , 634 (1966). J. Ito, "Synthesis of Calcio gadolinite", Amer. Mineral., <u>52</u> , 1523 (1967).
Be ₂ O ₄ Si(V ⁴⁺) Be ₂ SiO ₄ doped with V ⁴⁺	bright blue, 2 mm	slow cooling from V ₂ O ₅ or Ba ₃ (VO ₄) ₂ flux	precession and x-ray powder diffraction anal.		H. S. Peiser	C. Frondel and J. Ito, "Synthesis of the Scandium Ana- logues of Beryl", Amer. Mineral., <u>53</u> , 943 (1968).
Bi ₁₂ GeO ₂₀ GeBi ₁₂ O ₂₀		see Bi ₁₂ MO ₂₀	optical	see Bi ₁₂ Ga ₂ O ₂₁	A. Feldman D. Horowitz	
Bi ₁₂ MO ₂₀ 6Bi ₂ O ₃ ·MO ₂	M = Ge, Si, Ti; 0.5 x 1 cm	melt grown	see also Bi ₁₂ GeO ₂₀ , Bi ₃₂ Ga ₂ O ₅₁	Faraday rotation; piezoelectric electro-optic	W. S. Brower	E. L. Venturini, E. G. Spencer and A. A. Ballman, "Elasto- Optic Properties of Bi ₁₂ GeO ₂₀ , Bi ₁₂ SiO ₂₀ , Sr _{1-x} Ba _x Nb ₂ O ₆ , J. Appl. Phys., <u>40</u> , 1622 (1969).
7Bi ₂ O ₃ ·ZnO 17Bi ₂ O ₃ ·Ga ₂ O ₃	0.5 x 1 cm 0.5 x 1 cm	melt grown melt grown			W. S. Brower W. S. Brower	
Bi ₃₂ Ga ₂ O ₅₁ 16Bi ₂ O ₃ ·Ga ₂ O ₃		pulled from melt; prepd. by W. S. Brower	optical	optically active, high Faraday ro- tation; see also Bi ₁₂ GeO ₂₀	A. Feldman D. Horowitz	

Br ₂ bromine	from liquid at 20°C, 5Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	C. E. Weir, G. J. Piermarini, and S. Block, "Crystallography of Some High Pressure Forms of C ₆ H ₆ , CS ₂ , Br ₂ , CCl ₄ , and KNO ₃ ", J. Chem. Phys., 50, 2089 (1969)
CCl ₄	I structure from liquid at 20°C, 1Kb II structure from CCl ₄ -I at 80°C, 8Kb III structure from CCl ₄ -I at 120°C, 10Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br ₂ .
CS ₂	frozen from liquid at 20°C, 12Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br ₂ .
C ₅ FeN ₆ Na ₂ O·2H ₂ O Na ₂ Fe(CN) ₅ NO·2H ₂ O sodium nitroprusside	1 x 1 x 3 in. controlled growth from aq. soln.	Mossbauer spectroscopy	crystal structure, Standard Reference Material #725; Mossbauer spectroscopy	A. T. Horton J. J. Spilkerman, D. K. Snediker, F. M. Ruegg, and J. R. DeVoe, "Mossbauer Spectroscopy Standard for the Chemical Shift of Iron Compounds", NBS Misc. Publ. 260-13 (1967).	"Standard Reference Material for Mossbauer Spectroscopy", NBS Tech. News Bull., 50, 73 (May 1966).
C ₆ H ₆	I structure, see also SECTION II	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br ₂ .

$C_{10}H_{10}Cl_2CuN_2O_2$ Cu(C ₆ H ₅ NO) ₂ Cl ₂ with the following donants: Ba, Cd, Ni, ⁶¹ Ni (0.1-2%), Pb, and Zn bis(pyridine-N-oxide)copper (II) chloride	grown from methanol soln. 5 mm	optical and EPR spectrometry; chemical anal.	metal-metal interaction study	G. F. Kokoszka R. W. Duerst and G. F. Kokoszka, "Hyperfine Fields in Dimeric Coordination Complexes", J. Chem. Phys., <u>51</u> , 1673 (1969).
$C_{10}H_{10}Fe$ (C ₅ H ₅) ₂ Fe ferrocene	1 x 4 in	infrared and Raman spectrophotometry	infrared and Raman spectra vibrational transitions	A. T. Horton
$C_{12}H_{12}N_{10}NiO_6 \cdot 2H_2O$ Ni(C ₆ H ₆ N ₄) ₂ (H ₂ O) ₂ (NO ₃) ₂	$C_6H_6N_4$ (2,2'- bi-imidazole) + Ni(NO ₃) ₂ + H ₂ O	single crystal x-ray anal.	study of hydrogen bonding be- tween co- ordinated water and biimidazole molecules with nitrate groups	A. D. Mighell, C. W. Reimann, and F. A. Mauer, "The Crystal and Molecular Structure of Diaquo- bis(2,2'-Biimidazole) Nickel (II) Dinitrate, Ni(C ₆ H ₆ N ₄) ₂ (H ₂ O) ₂ (NO ₃) ₂ ", Acta Cryst., <u>B25</u> , 60 (1969).
$C_{12}H_{16}Br_2N_8Ni$ (C ₆ H ₅ N ₂) ₄ NiBr ₂ tetrakis (pyrazolo)- nickel (II) bromide	$C_6H_5N_2$ -(pyrazole) + NiBr ₂ + H ₂ O	x-ray anal., single crystal structure determ.; optical spectrophotometry	role of internal hydrogen bond in determ. elec- tronic energy ² levels of Ni ²⁺	A. D. Mighell, C. W. Reimann, and A. Santoro, "The Crystal and Molecular Structure of Di- bromotetrapyrazole Nickel (II)", Acta Cryst., <u>B25</u> , 595 (1969).
$C_{12}H_{16}Cl_2N_8Ni$ (C ₆ H ₅ N ₂) ₄ NiCl ₂ tetrakis(pyrazolo)- nickel (II) chloride	$C_6H_5N_2$ -(pyro- zole) + NiCl ₂ + H ₂ O			C. W. Reimann, A. D. Mighell, and F. Mauer, "The Crystal and Molecular Struc- ture of Tetrakis- pyrazolo-nickel Chloride, Ni(C ₆ H ₅ N ₂) ₄ Cl ₂ ", Acta Cryst., <u>23</u> , 135 (1967).

$C_{12}H_{18}$ $C_6(CH_3)_6$ hexamethyl- benzene	1" dia. x 3"	melt solidi- fication	x-ray, neutron diffractometry; differential thermal anal.	A. T. Horton	crystal structure; phase tran- sitions; lattice dynamics
$C_{12}H_{18}N_{24}Ni_3O_{18} \cdot 8H_2O$ $[(H_2O)_3(C_2H_5N_3)_3Ni]_2-Ni(NO_3)_6(H_2O)_2$		$C_2H_5N_3$ (1,2,4- triazole) + $Ni(NO_3)_2$ + H_2O	single crystal x-ray anal.	C. W. Reimann	C. W. Reimann and M. Zocchi, "The Structure of the Trinuclear Cation Bis-[u-(tri-1,2,4- triazolo-N ¹ ,N ²)- triaquonickel] nickel", Chem. Comm., 272 (1968).
$C_{12}H_{24}N_{14}NiO_6$ $(C_2H_4N_2)_6Ni(NO_3)_2$		$C_3H_4N_2$ (pyra- zole) + $Ni(NO_3)_2$ + H_2O	single crystal x-ray anal.	C. W. Reimann	C. W. Reimann, A. Santoro, and A. D. Mighell, "The Cry- stal and Molecular Structure of Hexa- pyrazolenickel- (II) Nitrate", $Ni(C_3H_4)_6WO_3 \cdot \frac{1}{2}H_2O$, Acta Cryst., B26, (1970).
$C_{18}H_{24}MN_{14}O_6$ $(C_3H_4N_2)_6Cd(NO_3)_2$ $(C_3H_4N_2)_6Co(NO_3)_2$ $(C_3H_4N_2)_6Ni(NO_3)_2$	M = Cd, Co, and Ni	$C_3H_4N_2$ (imida- zole) + $M(NO_3)_2$ + H_2O	single crystal x-ray anal.	C. W. Reimann	A. Santoro, A. D. Mighell, M. Zocchi, and C. W. Reimann, "The Crystal and Molecular Structure of Hexakis (Imida- zole) Nickel (II) Nitrate, $(C_3H_4N_3)_6$ $Ni(NO_3)_2$ ", Acta Cryst., B25, 842 (1969).

$C_{20}H_{42}$ n- $C_{20}H_{42}$ n-eicosane	2" dia. x 4"	melt solidi- fication	electrical, optical ab- sorption measurements	detm. free carrier mobility and G values; radiolysis measurements by use of optical ab- sorption techniques	A. T. Horton
CaF_2 also Gd-doped (0.1 to 1.0%)	1 x 3 x 5 mm	commercially obtained; cry- stals annealed in He + HF at 500-1200°C, rapidly quenched	absorption (200 to 2500 nm), EPR spectrometry; chemical anal. for Gd and accidental impurities	controlled point-defect equilibria studies; oxygen free	A. Franklin A. Franklin, "Mass Transport in Non- metallic Solids", Proc. of Mtg. of Basic Science Div. of British Ceramic Soc., London, Dec. 1969, in press. A. Franklin and S. Marzullo, "Orientation Kinetics of Gd^{3+} - Interstitial Pairs in CaF_2 ", J. Physics C, British Institute of Physics, to be published.
$CaGeO_5Ti$ $CaTiGeO_5$	1 x 1 x 2 mm	melt solidi- fication from stoichiometric composition	spectrochemical anal.; optical, x-ray diffrac- tion (powder and single crystal)	isostructural with synthetic CaO_5SiTi	C. R. Robbins C. R. Robbins, "Synthetic $CaTiSiO_5$ and its Germanium Analogue ($CaTiGeO_5$)", Mat. Res. Bull., 3, 693 (1968).
$CaMoO_4$	see under $MMoO_4$				

CaO ₅ SiTi	1 x 2.5 cm (1)	melt solidification (2), and Czochralski growth (1)	spectrochemical anal.; optical, x-ray diffractometry [powder and single crystal (2)]	serves as type structure for pure CaTiSiO ₅ (space group P ₂ /n); mineral with impurities, CaTiSiO ₄ (OH, F), has symmetry C ₂ /c; see also CaGeO ₅ Ti	C. R. Robbins W. S. Brower	(1) W. S. Brower and C. R. Robbins, "Growth of CaTiSiO ₅ by the Czochralski Method", J. Cryst. Growth, <u>3</u> , (1969). (2) See ref. under CaGeO ₅ Ti.
CaO ₄ W CaWO ₄	see under MO ₄ W					
Ca ₃ Fe ₂ MO ₁₂ Si ₂	M = Ti, Zr	slow cooling of Li ₂ MoO ₄ flux	powder and precession x-ray diffraction anal.	crystallographic sites detm. for both Ti and Zr in garnet structure	H. S. Peiser	J. Ito and C. Frondel, "Synthetic Zirconium and Titanium Garnets", Amer. Mineral., <u>52</u> , 773 (1967).
Ca ₃ TiFe ₂ Si ₂ O ₁₂	titanium garnet, dark brown, 2 mm also polycrystalline					
Ca ₃ ZrFe ₂ Si ₂ O ₁₂	zirconium garnet, chestnut brown, 0.55 mm, also polycrystalline					
CdF ₂ also with dopings	1 cm	Bridgman growth	electrical		W. R. Hosler W. S. Brower	P. Eisenberger and P. S. Pershan, "Electron Spin-Resonance and Infrared Studies of Semiconducting, Rare-Earth-Doped CdF ₂ ", Phys. Rev., <u>167</u> , 292 (1968).

CuCl	Vapor and Bridgman growth; prepd. by D. E. Roberts, also supplied by A. F. Armstrong AFRL, Bedford, Mass., and by A. Linz, Mass. Inst. Tech., Cambridge, Mass.	optical, dielectric	optical modulator	A. Feldman D. Horowitz	F. Sterzer, et al., "Cuprous Chloride Light Modulators", J. Opt. Soc. Am., <u>54</u> , 62 (1964). A. Feldman and D. Horowitz, "Refractive Index of CuCl", J. Opt. Soc. Am. <u>59</u> , 1406 (1969).
ClD ₄ N	3 x 3 x 13 mm see also H ₄ NX	NMR (D, ¹⁴ N) spectrometry	measurement of quadrupole coupling const. for D on ND ₄ ⁺ configuration; torsional motion of ND ₄ ⁺ ; crystal structure	R. A. Forman M. Linzer	M. Linzer and R. A. Forman, "NMR Studies of Single Crystal ND ₄ Cl", J. Chem. Phys., <u>46</u> , 4690 (1967).
ClO ₃ Na NaClO ₃	1 x 1 in.	optical microscopy (etch pits); x-ray topography	optical, x-ray measurements	A. T. Horton	R. D. Deslattes, B. G. Simson, A. T. Horton, "Solution Polishing of Oriented Single Crystals", Rev. of Sci. Inst., <u>36</u> , 944 (1965). R. Brooks, A. T. Horton, and J. L. Torgesen, "Occlusion of Mother Liquor in Solution-Grown Crystals", J. Cryst. Growth, <u>2</u> , 279 (1968).

<p>Cl₃CsPb</p> <p>TlCl also with dopants Cd, Pb, Nd, T</p>	<p>~ 2 mm, very soft</p>	<p>Bridgman growth; prepd. by D. E. Roberts</p>	<p>optical</p>	<p>piezo-optical luminescent properties</p>	<p>A. Feldman L. H. Grabner</p>	<p>R. Z. Bachrach and F. C. Brown. "Exciton Structure of TlCl and TlBr", Phys. Rev. Ltrs., 21, 685 (1968). A. Feldman, "Anoma- lous Splitting of the Exciton Peak in the Reflectivity of TlCl and TlBr", Bull. Am. Phys. Soc., <u>14</u>, 428 (1969). L. H. Grabner, "Edge Fluorescence in TlBr", Bull. Am. Phys. Soc., <u>14</u>, 131 (1969).</p>
<p>Cl₃CsPb</p> <p>CsPbCl₃</p>	<p>yellow, 1 cm. dia. x 2 cm.</p>	<p>grown by W. S. Brower, Bridgman method</p>	<p>dielectric constant</p>	<p>measured from 4.2K through phase trans- ition at 320K</p>	<p>K. F. Young</p>	<p>C. K. Moller, "Structure of Perovskite-like Ce Pb Trihalides", Kgel. Danske Videnskab. Selskab, Mat. Fys. Medd., 32, 1 (1959); also see Nature, <u>182</u>, 1436 (1958).</p>
<p>Cl₆K₂Re</p> <p>K₂ReCl₆</p>	<p>~ mm size</p>	<p>controlled evaporation, from soln.</p>	<p>ultra-sonic attenuation</p>	<p>second and higher order phase transitions</p>	<p>A. T. Horton</p>	
<p>CoM₂O₇Si₂</p> <p>Ca₂CoSi₂O₇</p> <p>Sr₂CoSi₂O₇</p>	<p>M = Ca, Sr; dark blue, 2 mm. Ca- and Sr cobalt akermanite; also poly- crystalline analogues with M = Cd, Cu, Fe, Mg, and Mn</p>	<p>slow cooling, flux Na₂WO₄</p>	<p>x-ray powder diffractometry</p>		<p>H. S. Peiser</p>	<p>J. Ito and H. S. Peiser, "Distorted Tetrahedra in Strontium Copper Akermanite", J. Res. NBS, <u>73A</u>, 69 (1969).</p>

$\text{CrH}_6\text{Mo}_6\text{Na}_3\text{O}_{24} \cdot n\text{H}_2\text{O}$ $\text{Na}(\text{CrMo}_6\text{O}_{24}\text{H}_6) \cdot n\text{H}_2\text{O}$	n = 8, prepn. leads to mm size crystals, no commercial supply available	aq. soln. of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O} + \text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ at pH = 4.5	complete single crystal x-ray structure anal.	readily dehydrates to a composition approximating $(\text{Na}_3(\text{CrMo}_6\text{O}_{24}\text{H}_6) \cdot 2\text{H}_2\text{O})$	A. Perloff (1) A. Perloff, "The Crystal Structure of Sodium Hexamolyb- dichromate (III) Octahydrate: $\text{Na}_3(\text{CrMo}_6\text{O}_{24}\text{H}_6) \cdot 8\text{H}_2\text{O}$ ", Inorg. Chem., in press. (2) A. Perloff, Doctoral Dissertation, Georgetown Univ. (1966) (3) G. A. Tsigidinos, Doctoral Dissertation, Boston Univ. (1961).
$\text{CrH}_6\text{NO}_6\text{S}_2 \cdot 12\text{H}_2\text{O}$ $\text{NH}_4\text{Cr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	n = 13, prepn. leads to mm size crystals, no commercial supply available	crystallizes on evaporation of aq. soln. of octahydrate at room temp.	complete single crystal x-ray structure anal.	readily dehydrates to a lower hydrate	(1) A. Perloff, paper in prepn.
$\text{CrH}_4\text{NO}_6\text{S}_2 \cdot 12\text{H}_2\text{O}$ $\text{NH}_4\text{Cr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	from aq. soln.	optical, magnetic	ferro- electric see also $\text{Al}_{n-1}\text{Cr}-\text{KOs}_2 \cdot 12\text{H}_2\text{O}$	G. A. Candela S. E. Stokowski*	
$\text{CuO}_4\text{S} \cdot 5\text{H}_2\text{O}$ $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	1 cm ²	controlled evaporation from soln.	NMR spectrometry	NMR spectro- scopy, wave guide experiments	A. T. Horton

Cu ₂ O	1.5 x 6 cm	grown from melt	optical and electrical measurements; air sensitive	W. S. Brower H. S. Parker	W. S. Brower and H. S. Parker, "Melt Growth of Large Cuprous Oxide", Single Crystals", Abstracts, ACCG- NBS Conf. on Crystal Growth, August 11-13, 1969, Washington, D. C., p. 115.
		prepd. by W. S. Brower	oxide- semiconductor dielectric constant	R. A. Forman W. R. Hosler K. F. Young	
F ₂ MgNa ₂ O ₄ Si ₆ M = Gd La Nd Pr Sm	largest crystal 2 x 2 x 6 cm colorless purple green yellow	flux (NaF) isothermal evaporation	x-ray powder diffraction anal.; visible emission spectrometry	H. S. Feiser	J. Ito, "Silicate Apatites and Oxya- patites", Amer. Mineral., <u>53</u> , 890 (1968).
F ₂ Mg MgF ₂	~ 1 cm ³	pulled from melt, D. E. Roberts	color centers, optical coatings	M. I. Cohen	R. F. Blunt and M. I. Cohen, "Irradiation- Induced Color Centers in Magnesium Fluoride", Phys. Rev., <u>153</u> , 1031 (1967).
F ₂ Pb PbF ₂ also Y, rare- earth dopings		Bridgeman growth		W. R. Hosler W. S. Brower	D. A. Jones, "Growth of Lead Fluoride Crystals from the Melt", Proc. Phys. Soc., <u>68B</u> , 165 (1955).

H ₂ O - VII ice VII	structure VII	from liquid at 100°C and 30 Kb	x-ray diffractometry	unit cell, space group, phase equilibrium	S. Block G. Piermarini C. E. Weir*	C. E. Weir, S. Block, and G. Piermarini, "Single Crystal X-ray Diffraction at High Pressures", J. Res. NBS, 69C, 275 (1965).
H ₄ NX NH ₄ X X = Br, Cl, I also with Tl doping	~ 1 cm ³	from aq. soln. by evaporation	optical	relation to alkali halides	R. A. Forman	
H ₆ NO ₄ P NH ₄ H ₂ PO ₄	3/4 x 3 in	controlled growth from soln.	optical microscopy (etch pits); x-ray topography	monochrometers, electro-optics, x-ray reference crystals	A. T. Horton	R. D. Deslattes, J. L. Torgesen, B. Paretzkin, and A. T. Horton, "Preliminary Studies on the Characterization of Solution-Grown ADP Crystals", Adv. in X-ray Anal., 8, p. 315 (1965).
"ADP"						R. D. Deslattes, J. L. Torgesen, B. Paretzkin, and A. T. Horton, "Observation of Dislocations in ADP: Production of Dislocation-Free Crystals", J. Appl. Phys., 37, 541 (1966).
InNaO ₆ Si ₂ NaInSi ₂ O ₆	colorless indium agerine, 1 cm; also obtained as polycrystalline	slow cooling from Na ₂ WO ₄ or Na ₂ MoO ₄ flux	powder and precession x-ray diffraction anal.	see also Na ₆ ScSi ₂	H. S. Peiser	J. Ito, "Synthetic Indium Silicates and Hydrogarnet", Am. Mineral., 53, 1663 (1968).

KNO ₃ III	structure III	obtained from KNO ₃ II at elevated temp. and pressure	x-ray diffractometry	detm. unit cell, space group, phase equilibrium	S. Block G. Piermarini C. E. Weir*	See ref. under Br ₂ .
KNO ₃ IV	structure IV	from saturated aq. soln. KNO ₃ at 100°C, 20 Kd				
KO ₃ Ta		pred. by W. S. Brower; also obtained with doping from A. Linz, Mass. Inst. Tech., Cambridge, Mass.; flux growth	electrical, optical	oxide semiconductor	M. I. Cohen W. R. Hosler	M. I. Cohen, <u>et al.</u> , "Lattice Absorption in Strontium Titanate", Bull. Am. Phys. Soc., <u>11</u> , 498 (1969).
KTaO ₃ , also doped with Mn						W. R. Hosler and H. P. R. Frederikse, "Magnetoresistive Effects in KTaO ₃ ", Sol. State Comm., <u>7</u> , 1443 (1969).

MnMoO ₄	M = Ca, Pb, Sr	1 x 3 cm	melt grown	x-ray topography; dielectric constant, etch pits	see also MnMoO ₄	W. S. Brower and P. H. Fang, "Dielectric Constants of PbMoO ₄ and CaMoO ₄ ", Phys. Rev., <u>149</u> , 640 (1966).
CdMoO ₄ PbMoO ₄ SrMoO ₄					acousto- optic devices	W. S. Brower W. S. Brower and P. H. Fang, "Dielectric Constants of PbWO ₄ and CaWO ₄ ", J. Appl. Phys., <u>38</u> , 2391 (1967).
				mechanical	elastic constants of CaMoO ₄	J. B. Wachtman, Jr., W. S. Brower, Jr. and E. N. Farabaugh, "Elastic Constants of Single Crystal Calcium Molybdate (CaMoO ₄)", J. Am. Ceram. Soc., <u>51</u> , 341 (1968).
MO ₄ W	M = Ca, Pb, Sr, Zn; 1 x 3 cm		melt grown	x-ray topography; dielectric constant; etch pits	acousto- optic devices	W. A. Bonner, "Growth of Large Single Crystal Lead Molyb- date for Acousto- Optic Applications", Abstr. ACCG-NBS Conf., August, 1969, p. 116.
CaWO ₄ PbWO ₄ SrWO ₄ ZnWO ₄						
ZnWO ₄	1 cm			optical	magneto- optical properties	R. F. Blunt See refs. under MnMoO ₄ .

MnMoO ₄	1 cm	pulled from melt; prepd. by D. E. Roberts	optical	magneto-optical properties	R. F. Blunt	See also under MnO ₄ W and MO ₄ W for additional refs. to Scheelite crystals.
MnO ₄ W MnWO ₄	1 cm	pulled from melt; prepd. by D. E. Roberts.	optical	magneto-optical properties	R. F. Blunt	See also MO ₄ W.
MoO ₄ Pb MoO ₄ Sr	see MMoO ₄					
NaO ₆ ScSi ₂ NaScSi ₂ O ₆	colorless, scandium aegirine, 0.5 cm; also as poly-crystalline	slow cooling from Na ₂ WO ₄ or Na ₂ MoO ₄ flux	powder and precession x-ray diffraction anal.	see also InNaO ₆ Si ₂	H. S. Peiser	J. Ito and C. Frondel, "Synthesis of Scandium Analogues of Aegirine, Andradite and Melanotekite", Am. Mineral., <u>53</u> , 1276 (1968).
NiTi TiNi	wire	obtained from NOL, White Oak, Md.	electrical and magnetic	exhibits martensitic transition; i.e., shows mechanical memory through transition	W. R. Hosler	F. E. Wang, B. F. DeSavage, W. J. R. Buehler, and W. R. Hosler, "The Irreversible Critical Range in the TiNi Transition", J. Appl. Phys. <u>39</u> , 2166 (1968).
O ₂ Th ThO ₂	2 x 1 x 1 cm	selected from arc melt by G. R. Findlay, Norton Co.	Laue diffraction	elastic constants	J. Wachtman, Jr.	P. B. Macedo, W. Capps, and J. B. Wachtman, Jr., "Elastic Constants of Single Crystal ThO ₂ at 25°C", J. Am. Ceram. Soc., <u>47</u> , 651 (1964).

TiO ₂	single crystal rod	Verneuil process, reheated in η_0	compressibility to 10 Kbar	C. E. Weir*	See ref. under Al ₂ O ₃ .
TiO ₂ also with dopants: Al, Cr, Cu, Fe, Mo, Nb, rare earths, V, W, Zn	1 cm	flame fusion, prepd. by D. E. Roberts	IR spectrometry; electrical, optical	R. F. Blunt T. Chang T. C. Ensign* W. R. Hosler	T. Chang, "Electron Spin Resonance of Mn ²⁺ in Rutile", Phys. Rev., <u>136</u> , A413 (1964); "Paramagnetic Resonance Spectrum of W ⁵⁺ in Rutile (TiO ₂)", Phys. Rev., <u>147</u> , 264 (1966).
TiO ₂ pure, also with dopants: Cr, Ni; oxidized and reduced conditions	single crystal rod	flame fusion	Laue diffraction patterns	J. Wachtman, Jr.	H. P. R. Frederikse, "Recent Studies on Rutile", J. Appl. Phys. Suppl., <u>32</u> , 2211 (1961).
			elastic constants; internal friction	W. E. Tefft, and D. G. Lam, Jr., "Elastic Constants of Rutile (TiO ₂)", J. Res. NBS, <u>66A</u> , 465 (1962).	
				S. Spinner and J. B. Wachtman, Jr., "Some Elastic Compliances of Single Crystal Rutile from 25 to 1000°C", J. Res. NBS, <u>68A</u> , 669 (1964).	
				J. B. Wachtman, Jr. and L. R. Doyle, "Internal Friction in Rutile Containing Point Defects", Phys. Rev., <u>135</u> , A276 (1964).	
				J. B. Wachtman, Jr., S. Spinner, W. S. Broer, T. Fridinger, and R. W. Dickson, "Internal Friction in Rutile Containing Ni or Cr", Phys. Rev., <u>148</u> , 811 (1966).	

O ₂ U	3 x 1 x 1 cm	selected from arc melt by H. J. Anderson of Hanford Atomic Products Operation	density, coulometric titration, spec. analysis, Laue diffraction	elastic constants	J. Wachtman, Jr. J. B. Wachtman, Jr., M. L. Wheat, H. J. Anderson and J. L. Bates, "Elastic Constants of Single Crystal UO ₂ at 25°C", J. Nuclear Materials, <u>16</u> , 39 (1968).
O ₃ PbSi PbSiO ₃	alamosite, colorless, 2 mm; also prepd. as polycrystal- line	hydrothermal, spontaneous nucleation isothermal	x-ray structural anal. (M. Mydlar, Ceramic Engr. Dept., Univ. of Mo.)	structure	J. Ito, "Synthesis of Some Lead Calcium Zinc Silicates", Amer. Mineral., <u>53</u> , 231 (1968).

0.5w1	1 cm	flame fusion; also supplied by D. Beals, National Lead Co., South Amboy, N. J.	optical, electrical; EPR spectrometry	insulator; semiconductor, super- conductor; pseudo- ferroelectric; photochromic	T. Chang J. Levendecker, "Electronic Energy Bands in Strontium Titanate", Phys. Rev., 135, A1321 (1964). H. P. R. Frederikse, W. R. Hosler, and W. R. Thurber, "Experi- mental Evidence Concerning the Conduction and Band of SrTiO_3 ", J. Phys. Soc. Japan, 21, Suppl., 32 (1966). J. F. Schooley and W. R. Thurber, "Super- conductivity in Semiconducting SrTiO_3 ", J. Phys. Soc. Japan, 21, Suppl., 639 (1966). A. Feldman and D. Horowitz, "Stress- Induced Dichroism at the Absorption Edge of Strontium Titanate", Sol. State Comm., 6, 607 (1968). C. R. Robbins, "Growth of Strontium Titanate from a Silica Flux", J. Cryst. Growth, 2, 402 (1968).
SrTiO_3	1 - 2.5 mm	melt solidi- fication using SiO_2 as flux	spectrochemical, optical and x-ray powder diffracto- metry	prepd. in absence of fluorides, etc.	C. R. Robbins

NBS STAFF MEMBERS

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FOR ADDITIONAL
INFORMATIONREFERENCES (Author,
Title, Journal)

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
Ag	high-purity rod, 1/4 in. dia. x 2-1/2 in.	special prepn. for Office of Standard Reference Materials, SRM # 748	Knudsen, torque Knudsen, and mass spectrometric detm. of vapor pressure as function of temperature; detm. heat of sublimation	calibrn. of vapor pressure apparatus and techniques (10-8 to 10-3 atm., 1000-1600°K); this and other elements (see Au, Cd, Pt, and W) will each cover a different temperature range; these five materials cover 400-3000°C	R. C. Paule	R. C. Paule and J. Mandel, NBS Special Publication # 260.
AgNO ₃ "high-AgNO ₃ "	polycrystalline surface, α50μ	high temperature x-ray diffractometer furnace	x-ray powder diffractometry	unstable at room temp.	E. M. Levin	E. M. Levin, "X-Ray Determination of the Thermal Expansion of Silver Nitrate", J. Am. Ceram. Soc., 52, 53 (1969).
AlN aluminum nitride	100-300 mesh	four commercial sources	x-ray diffractometry	parent compound for study of transport chemistry	F. E. Brinckman	
Al ₂ BaO ₄ BaAl ₂ O ₄		γ-Al ₂ O ₃ + BaCO ₃ at 1200°C for 1.5 hr.	x-ray diffractometry; petrographic microscopy		H. Swanson J. deGroot H. McMurdie	H. Swanson et al., "Standard X-Ray Diffraction Powder Patterns", NBS Monograph No. 25, Sect. 5, p. 11 (1967).

See ref. above.

Al ₂ O ₃	See also IV. SINGLE CRYSTALS	(a) commercial powder and crystals (b) powder purified by solar furnace	melting point detm.	S. J. Schneider and C. L. McDaniel, "Effect of Environment Upon the Melting Point of Al ₂ O ₃ ", J. Res. NBS, 71A, 317 (1967).
				S. J. Schneider, "Cooperative Deter- mination of the Melting Point of Al ₂ O ₃ ", to be publish- ed in Pure and Applied Chemistry (1970).

Au	high purity wire, 0.055 in. dia. x 6 in. Standard Reference Material # 745	special prepn. for Office of Standard Reference Materials, SRM # 745	see under Ag	R. C. Paule temp. range 1300-2100°K; see also under Ag	R. C. Paule and J. Mandel, "Analysis of Interlaboratory Measurements on the Vapor Pressure of Gold", NBS Special Publication # 260- 19.
	0.055" wire SRM # 745	available as SRM # 745	see under Ag	see under Ag	See ref. above.

BF ₄ M KBF ₄ NaBF ₄	M = K, Na; obsd. also in aq. soln.	commercial	NMR (11B, 19F) spectrometry	model com- pounds for high resolution and broad- line NMR; JB-F as related to solvents	F. E. Brinckman T. C. Farrar
BH ₃ O ₃ H ₃ BO ₃		commercial	compressibility to 10 Kb	see also Al ₂ O ₃ under Section IV.	C. E. Weir, "Compress- ibility of Eleven In- organic Materials", J. Res. NBS, 69A, 29 (1965).

BH ₄ M	M = Li, K, Na; obsd. also in aq. soln.	commercial	NMR (¹ H, ¹¹ B) spectrometry	model com- pounds, detm. phase trans- itions and motions in solids	T. C. Farrar	T. C. Farrar and T. Tang, "A Nuclear Magnetic Resonance and Relaxation Study of Dimethoxyborane", J. Res. NBS, <u>73A</u> , 135 (1969).
B ₂ O ₃	~50μ part- icles, calcite type	solid state rxn. of Sc ₂ O ₃ + B ₂ O ₃ in sealed tube	x-ray powder diffractometry, polarizing microscopy	phase diagram	E. M. Levin	E. M. Levin, "System Sc ₂ O ₃ -B ₂ O ₃ ", J. Am. Ceram. Soc., <u>50</u> , 53 (1967).
B ₂ O ₃		controlled dehydration of H ₃ BO ₃	compressibility to 10 Kb		C. E. Weir*	See ref. under BH ₃ O ₃ .
B ₂ O ₃ · 3Nb ₂ O ₅ "3Nb ₂ O ₅ · B ₂ O ₃ "	~50μ	solid state rxn. of B ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tube	x-ray powder diffractometry	phase diagram	E. M. Levin	E. M. Levin, "Phase Equilibria on the System Niobium Pentoxide-Boric Oxide", J. Res. NBS, <u>70A</u> , 11 (1966).
Borate minerals, anhydrous	~80 in number, fine powders	metal oxide + H ₃ BO ₃ at elevated temp.	infrared (2000- 300 cm ⁻¹) spectro- metry	correlation of anion and spectra	C. E. Weir*	C. E. Weir and R. A. Schroeder, "Infrared Spectra of the Crystalline Inorganic Borates", J. Res. NBS, <u>68A</u> , 465 (1965).
Borate minerals, hydrated	~42 natural and synthetic	obtained from the Smithsonian Inst. U. S. Geological Survey, Wash- ington, D. C. and Menlo Park, Calif.	infrared spectro- metry, principally in 2000-300 cm ⁻¹ region	evaluation of limita- tions of spectra in identification, effects of treatment on spectra	C. E. Weir*	C. E. Weir, "Infrared Spectra of the Hydrated Borates", J. Res. NBS, <u>70A</u> , 153 (1966).
BaF ₃ Li LiBaF ₃		fuse stoichi- ometric amt. LiF + BaF ₂ at 800°, annealed at 500° to re- move BaF ₂ ppt.	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 35.

<p> $\text{BaGe}_3\text{O}_9\text{Ti}$ $\text{BaTiGe}_3\text{O}_9$ </p>	<p>20-40μ</p>	<p> solid state rxn. of BaTiO_3 + GeO_2 in sealed Pt tube at 1160°C </p>	<p> optical and x-ray powder diffractometry </p>	<p> stable from 1132 to 1235°C but readily obtainable by quenching to ambient temp.; serves as a structural intermediate between BaGe_4O_9 and $\text{BaTiSi}_3\text{O}_9$ </p>	<p>C. R. Robbins</p>	<p> C. R. Robbins, "The Compound $\text{BaTiGe}_3\text{O}_9$", J. Am. Ceram. Soc., <u>41</u>, 610 (1968). </p>
<p> $\text{Be}_2\text{CuO}_{10}\text{Si}_2\text{Y}_2$ $\text{CuY}_2\text{Be}_2\text{Si}_2\text{O}_{10}$ </p>	<p> polycrystalline for all possible combinations with $\text{MLn}_2\text{Be}_2\text{Si}_2\text{O}_{10}$, where M=Mg, Ni, Zn, Co, Cu, Fe, Mn, and Cd; single crystal (1mm) also studied </p>	<p> slow cooling of solute in Na_2WO_4 or $\text{Na}_2\text{W}_2\text{O}_7$ flux </p>	<p> precession and powder x-ray diffractometry </p>		<p>H. S. Peiser</p>	<p> J. Ito, "The Synthesis of Gadolinite", Proc. Jap. Acad., <u>41</u>, 404 (1965). J. Ito, "A Note on the Gadolinite Synthesis", Proc. J. Acad., <u>42</u>, 634 (1966). J. Ito, "Synthesis of Calciogadolinite", Amer. Mineral., <u>52</u>, 1523 (1967). </p>
<p> BiNbO_4 </p>	<p> see also: BiO_4Ta BiO_4V NbO_4Sb O_4SbTa O_4SbV </p>	<p> solid state rxn. of Bi_2O_3 + Nb_2O_5 in sealed Pt tubes at 950 - 1250°C </p>	<p> x-ray powder diffractometry </p>		<p>R. S. Roth</p>	<p> R. S. Roth and J. L. Waring, "Synthesis and Stability of Bismutotantalite, Stibiotantalite, and Chemically Similar ABO_4 Compounds", Am. Mineral., <u>48</u>, 1348 (1963). </p>
<p> BiO_4Ta BiTaO_4 </p>		<p> solid state rxn. of Bi_2O_3 + Ta_2O_5 in sealed Pt tubes at 845 - 1150°C </p>	<p> see BiNbO_4 </p>		<p>R. S. Roth</p>	<p> See ref. under BiNbO_4. </p>
<p> BiO_4V BiVO_4 </p>		<p> solid state rxn. of Bi_2O_3 + V_2O_5 in sealed Pt tubes at 500-1000°C </p>	<p> see BiNbO_4 </p>		<p>R. S. Roth</p>	<p> See ref. under BiNbO_4. </p>

Bi_2O_3	cubic	high-temp. x-ray diffractometer furnace; programmed heating and cooling	differential thermal anal.; x-ray diffractometry	obsd. at room temp. as metastable phase	E. M. Levin S. Roth, "Polymorphism of Bismuth Sesquioxide, I. Pure Bi_2O_3 ", J. Res. NBS, 68A, 189 (1964); <u>IBid.</u> , II, 197-206.
tetragonal					
$\text{Br}_2\text{O}_2\text{Pb}_3$ $\text{Pb}_3\text{O}_2\text{Br}_2$		heat stoichiometric PbO (red) + PbBr_2 in Au boat, in vacuo, at 350°C for 45 min.	x-ray diffractometry, petrographic microscopy		E. M. Levin J. deGroot H. McMurdie See ref. under Al_2BaO_4 , p. 32.
Br_3Ti TiBr_3	dark purple, fine powder or small crystals; see also Cl_3Ti	$\text{TiBr}_4 + \text{H}_2 \rightarrow$ using hot Ir filament	elemental anal. for Ti, Br	sublimes at $\sim 500^\circ\text{C}$ with disproportionation, strong reducing agent; catalyst for olefin polymerization; air/moisture sensitive	R. B. Johannesen J. M. Sherkey, "Titanium-(III) Chloride and Titanium (III) Bromide", Inorg. Synth., 6, 57 (1960).
Br_4Ti TiBr_4	yellow, m.p. 38°C	$\text{TiCl}_4 + \text{HBr} \rightarrow$ at b.p. of $\text{TiCl}_{4-n}\text{Br}_n$	elemental anal., m.p., b.p.	strong Lewis Acid properties, air/moisture sensitive	R. B. Johannesen G. L. Gordon, "Titanium (IV) Bromide", Inorg. Synth., 9, 46 (1967).
$\text{Br}_6\text{H}_8\text{N}_2\text{Os}$ $(\text{NH}_4)_2\text{OsBr}_6$	mixed crystals (solid soln.) of $(\text{NH}_4)_2(\text{Os-Pt})\text{Br}_6$; also prepd where $\text{Os}/\text{Os} + \text{Pt} = 0.0763$	(a) $\text{OsO}_4 + \text{HBr(aq)}$ (b) (a) + NH_4Br	elemental anal., x-ray diffractometry	parent compounds for magnetic susceptibility studies	R. B. Johannesen G. A. Candella, "Magnetic Susceptibilities and Dilution Effects in Low-Spin d^4 Complexes: Osmium (IV)", Inorg. Chem., 2, 67 (1963).

C_2Si	cubic, ~200 mesh	Si + C above m.p. of Si	x-ray diffractometry, vapor pressure	other polymorphs present, C-contaminated	E. R. Plante
$C_2Na_2O_4$ $Na_2C_2O_4$ sodium oxalate	NBS standard and sample No. 40d	NBS standard and sample No. 40d	assay 99.9% $Na_2C_2O_4$; x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie
C_3Al_4	large crystallites embedded in polycrystalline matrix	Al(1) + C at 1400°C	x-ray diffractometry, vapor pressure	usually contains excess C; moisture sensitive	E. R. Plante and C. H. Schreyer, "Dissociation Pressure of Aluminum Carbide Using a Rotating Knudsen Cell", J. Res. NBS, <u>70A</u> , 253 (1966).
C_4HCoO_4 $HCo(CO)_4$	unstable liquid, studied as solid	see SECTION II	see SECTION II	see SECTION II.	T. C. Farrar
$C_6H_{12}F_2O_4W$ $(CH_3O)_4WF_2$	colorless crystals m.p. 81-83°C cis-stereoisomer only	WF_6 + $4CH_3OSi(CH_3)_3$ in C_6Fe	NMR(1H , ^{19}F) mol. wt. elemental analysis	one of a series of stable tungsten (VI) compounds; easily purified by sublimation; cis-difluoro configuration; provides basis for W (VI) stereochemistry; air/moisture sensitive.	L. B. Brinckman F. E. Brinckman L. B. Handy* istry of the Methoxyfluorotungsten (VI) Series", Chem. Comm., 214 (1970).

$C_6H_{10}CuO_4 \cdot H_2O$ Copper (II) propionate monohydrate	also Zn- doped	heat $Cu(Zn)CO_3$ + $[CH_3CH_2CO]_2O$ + H_2O	EPR spectrometry	model com- pounds for determ. magnetic parameters in dimeric $Cu(II)$ alkanoates from EPR studies of polycrystalline samples	M. Linzer	G. F. Kokoszka, M. Linzer, G. Gordon, "Electron Paramagnetic Resonance Spectra of Polycrystalline Dimeric Complexes. Copper Propionate Monohydrate and Zinc- Doped Copper Pro- pionate Monohydrate", Inorg. Chem., <u>7</u> , 1730 (1968).
$C_6H_{18}HgSi_2$ [$(CH_3)_3Si]_2Hg$	yellow	$(CH_3)_3SiX +$ Na/Hg at 55°C (X=Cl, Br) (1)	NMR (1H) spectrometry	source of trimethyl- silyl group or radical (2); toxic, volatile Hg compound, soln. light- sensitive	F. E. Brinckman L. B. Handy*	(1) E. Wiberg, et. al, "Recent Developments in the Chemistry of Metal Silyls of the Type $M(SiR_3)_n$ ", Angew. Chem. internat. Edit., <u>2</u> , 507 (1963). (2) C. Eaborn, R. A. Jackson, and R. W. Walsingham, "The Reduction of Organo- silicon Radicals in Solution", Chem. Comm., <u>1965</u> , 300.
$C_{12}H_{36}LiN_2NaSi_4$ [$(CH_3)_3Si]_2N \cdot Li^+Na^-$ > $[M(Si(CH_3)_3)_2$		metallation of N-H on disilazanes; sample supplied by Prof. U. U. Wannagat, I. U. Carolo-Wilhelmina, Braunschweig, Germany	see ref., NMR ($^1H, ^7Li$, 14N , ^{23}Na) spectrometry	first ex- ample of NMR measure- ment of a mixed dimeric electron- deficient com- pound in the solid state	M. Linzer	D. Mootz, A. Zinnius, and B. Botcher, "Association in the Solid State of Bis(trimethylsilyl) amidolithium and Methyltrimethyl- silylanatoberyllium", Angew. Chem., <u>81</u> , 398 (1969).
$C_{20}H_{16}Cu_2N_2O \cdot H_2O$ $Cu_2(C_5H_4N_5)_4 \cdot$ H_2O		$Cu(ClO_4)_2(aq)$ + adenine, adjust pH	chemical anal., optical and EPR spectroscopy	metal-metal bond inter- actions pertinent to biochemical processes involving nucleic acids	G. F. Kokoszka	R. W. Duerst, S. J. Baum, and G. F. Kokoszka, "Exchange Coupling in Two Dimeric Copper Adenine Complexes", Nature, <u>222</u> , 665 (1969).
$C_{20}H_{20}Cl_4Cu_2N_2O_{16}$ $\cdot 3H_2O$ $Cu_2(C_5H_5N_5)_4 \cdot$ $(ClO_4)_4 \cdot 3H_2O$	$(C_5H_5N) =$ adenine moiety					

CaCl ₂ ·6H ₂ O CsCaCl ₃	CsCl + CaCl ₂ at ~ 900°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 21.
CaNa ₂ O ₈ S ₂ Na ₂ Ca(SO ₄) ₂	CaCl ₂ +Na ₂ SO ₄ in aq. soln.; intermediate CaSO ₄ forms, desired product after several hr., washed with alcohol	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 59.
Cd	high purity rod, 1/4 in. dia. x 2-1/2 in.	special prepr. for Office of Standard Reference Materials; SRM # 746	temp. range 400-600°K; see also under Ag	See ref. under Ag.
CdCl ₂ ·Cs CsCdCl ₃	stoichiometric CsCl+CdCl ₂ fusion	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 19.
CdCl ₂ ·H ₄ N NH ₄ CdCl ₃	stoichiometric NH ₄ Cl+CdCl ₂ (aq. soln.) + ppt.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 6.
CdCl ₂ ·K KCdCl ₃	fuse KCl + CdCl ₂ ·2.5H ₂ O at 550°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 38.
CdCl ₃ ·Rb	orthorhombic tetragonal	ppt. from aq. soln. RbCl + CdCl ₂ fuse RbCl + CdCl ₂ (anhydr.) at ~ 500°C	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 41. Same, p. 43.

CdCr_2O_4	(a) $\text{CdO} + \text{Cr}_2\text{O}_3$ heated in vacuo at 300°C for 17 hr. (b) ground, pelletized, heated at 1050°C for 1.5 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 16.
$\text{CdO}_4\text{S} \cdot \text{H}_2\text{O}$ $\text{CdSO}_4 \cdot \text{H}_2\text{O}$	(a) crystallized from aq. soln. at 95°C ; (b) also prepd. from CdSO_4 or $\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ on prolonged exposure to air of RH = 50%.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 10.
$3\text{CdO}_4\text{S} \cdot 8\text{H}_2\text{O}$	obtained from Johnson, Matthey & Co., Ltd.	high humidity necessary to prevent formation of monohydrate		same, p. 8.
ClH_4NO $\text{NH}_3\text{OHC1}$	obtained from Naval Ordnance Plant, Indian Head, Md.	NMR (^1H) spectrometry, vapor pressure	T. C. Farrar	
Cl_2P PCl_2 (free radical)	at 77K in PCl_3 matrix, C_{2v} symmetry $\text{PCl}_2 + \text{PCl}_4 + \text{Cl}$	EPR spectrometry	F. E. Brinckman G. F. Kokoszka	G. F. Kokoszka and F. E. Brinckman, "Electron Paramagnetic Resonance Studies of Phosphorus-Containing Reactive Intermediates", Chem. Comm., 349 (1969).
Cl_4P PCl_4 (free radical)	C_{2v} symmetry	formation of PCl_4 by sequential rxns.; evaluation of molecular bonding, relation to chlorophosphorylation via radical intermediates	G. F. Kokoszka and F. E. Brinckman, "Electron Paramagnetic Resonance Studies of Phosphorus-Containing Reactive Intermediates", J. Am. Chem. Soc., 92, 1199 (1970).	

Cl₃CuCl₃
CsO₂Cl₃

co-pptd. CsCl + CoCl₂ heated in sealed glass tube at 500°C

H. Swanson
J. deGroot
H. McMurdie

See ref. under
C₂Na₂O₄, p. 11.

Cl₃CoH₄N
NH₄CoCl₃

co-pptd. NH₄Cl + CoCl₂ heated in sealed glass tube at 500°C

H. Swanson
J. deGroot
H. McMurdie

See ref. under
C₂Na₂O₄, p. 5.

Cl₃CoRb
RbCoCl₃

co-pptd. RbCl + CoCl₂ heated in sealed glass tube at 500°C

H. Swanson
J. deGroot
H. McMurdie

See ref. under
C₂Na₂O₄, p. 57.

Cl₃CsCu
CsCuCl₃

dark red hex. prisms terminated by bipyramids

x-ray diffractometry, petrographic microscopy

H. Swanson
J. deGroot
H. McMurdie

See ref. under
Al₂BaO₄, p. 22.

Cl₃CsNi
CsNiCl₃

co-pptd. CsCl + NiCl₂ heated in sealed glass tube at 500°C

x-ray diffractometry, petrographic microscopy

H. Swanson
J. deGroot
H. McMurdie

See ref. under
C₂Na₂O₄, p. 12.

Cl₃CsPb
CsPbCl₃

fuse CsCl + PbCl₂ at ~ 500°C

x-ray diffractometry, petrographic microscopy

H. Swanson
J. deGroot
H. McMurdie

See ref. under
Al₂BaO₄, p. 24.
See also in
SECTION IV.

Cl₃CsSr
CsSrCl₃

SrCl₂ + CsCl melted at 900°C

x-ray diffractometry, petrographic microscopy

H. Swanson
J. deGroot
H. McMurdie

See ref. under
C₂Na₂O₄, p. 13.

Cl₃H₄HgN
NH₄HgCl₃

pptd. from soln. of NH₄Cl + HgCl₂

x-ray diffractometry, petrographic microscopy

H. Swanson
J. deGroot
H. McMurdie

See ref. under
Al₂BaO₄, p. 9.

$\text{Cl}_3\text{H}_4\text{NNi}$ NH_4NiCl_3	(a) $\text{NiCl}_2 \cdot 2\text{H}_2\text{O} + \text{HCl}$ at $150^\circ\text{C} + \text{NiCl}_2$ (b) (a) + NH_4Cl in sealed glass tube at 300°C for 72 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 6.
$\text{Cl}_3\text{HgNa} \cdot 2\text{H}_2\text{O}$ $\text{NaHgCl}_3 \cdot 2\text{H}_2\text{O}$	crystallization from aq. soln. of equimolar amts of $\text{NaCl} + \text{HgCl}_2$	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 66.
Cl_3NiRb RbNiCl_3	co-pptd. $\text{RbCl} + \text{NiCl}_2$ heated in sealed glass tube at 500°C	moderately hygroscopic	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 58.
Cl_4P	see Cl_2P	see Cl_2P		
CoF_3K KCoF_3	pptd. from mixt. $\text{KF} + \text{CoF}_2$ soln., washed	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 37.
$\text{CoNa}_2\text{O}_8\text{S}_2 \cdot 4\text{H}_2\text{O}$ $\text{Na}_2\text{Co}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$	crystallized from aq. soln. $\text{Na}_2\text{SO}_4 + \text{CaSO}_4$ at room temp.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 61.
CoO_6Sb_2 CoSb_2O_6	$\text{CoC}_2\text{O}_4 + \text{Sb}_2\text{O}_5$ at 1000°C for 30 min. in Au boat	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 26.
$\text{Co}_2\text{K}_2\text{O}_{12}\text{S}_3$ $\text{K}_2\text{Co}_2(\text{SO}_4)_3$	$\text{K}_2\text{SO}_4 + \text{CoSO}_4$ melted at 600°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 35.

CuF ₃ K		add CuCl ₂ to excess KF in soln. → ppt.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 38.
KCuF ₄					
Cu ₆ Sb ₂		solid state rxn. of CuO + Sb ₂ O ₅ in Ag boat at 945°C in vacuo for 2 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 27.
CuSb ₂ O ₆					
ErO ₄ V		Er ₂ O ₄ + V ₂ O ₅ heated at 1100°C for 30 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 29.
ErVO ₄					
FMPO ₃					
BaPO ₃ F	also studied in soln.	Na ₂ PO ₃ F + MC ₂ ; M = Ba, Ca, K	x-ray diffractometry, NMR (19F, 31P) spectrometry	T. C. Farrar A. Perloff D. VanderHart	D. L. VanderHart, H. S. Gutowsky, and I. C. Farrar, "NMR Study of BaPO ₃ : 31P and 19F Chemical Shift Anisotropies and the Absolute Sign of the F-P Coupling Constant", J. Chem. Phys., <u>50</u> , 1050 (1969).
CaPO ₃ F					
K ₂ PO ₃ F					
Na ₂ PO ₃ F					
F ₃ Fek		aq. soln. FeCl ₂ + KF → ppt., washed, heated to 400°C in <u>vacuo</u>	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 39.
KFeF ₃					
F ₃ KMG		HF + slurry of K ₂ CO ₃ + MgCO ₃ , evaporated to dryness; x-ray pattern sharpened by heating sample to m.p.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 42.
KMG ⁺ F ₃					

F ₃ KMn KMnF ₃	aq. MnCl ₂ + excess KF in soln. → ppt.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 45.
F ₃ MnNa NaMnF ₃	aq. MnCl ₂ + excess KF in soln. → ppt.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 65.
F ₃ NaZn NaZnF ₃	soln. ZnCl ₂ + conc. soln. NaF + ppt., washed, annealed at 500°C	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 74.
FeNbO ₄	solid state rxn. of Fe ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tube at 1000- 1475°C	x-ray powder diffractometry	R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Ixiolite and Other Polymorphic Types of FeNbO ₄ ", Am. Mineral., <u>49</u> , 242 (1964).
GdO ₄ V GdVO ₄	Gd ₂ (C ₂ O ₄) ₃ + V ₂ O ₅ at 800°C for 15 min.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 30.
GeO ₂ •9Nb ₂ O ₅	solid state rxn. in sealed Pt tube	x-ray powder diffractometry	E. M. Levin	E. M. Levin, "Phase Equilibria in the System Niobium Pentoxide-Germanium Dioxide", J. Res. NBS, <u>70A</u> , 5 (1966).
Ge ₄ O ₃ Pb PbGe ₄ O ₉	solid state rxn. of PbO + GeO ₂ in sealed Pt tube at 700°C	optical and x-ray powder diffracto- metry	C. R. Robbins with BaGe ₄ O ₉ SrGe ₄ O ₉	C. R. Robbins and E. M. Levin, "Tetra- germanates of Strontium Lead and Barium of Formula Type AB ₄ O ₉ ", J. Res. NBS, <u>65A</u> , 127 (1961).

BaO, Sr SrGe_2O_7	solid state rxn. of $\text{SrO} + \text{GeO}_2$ in sealed Pt tube at 1200°C	specimen and x-ray powder diffractometry	isostructural with $\text{K}_2\text{Ge}_2\text{O}_7$, PbGe_2O_7	C. K. Watkins See ref. under Ge_2O_7 lib.
$\text{H}_2\text{M}_{10}\text{O}_{26}\text{P}_6$ $\text{Ba}_{10}\text{P}_6\text{O}_{24}(\text{OH})_2$ $\text{Pb}_{10}\text{P}_6\text{O}_{24}(\text{OH})_2$ $\text{Sr}_{10}\text{P}_6\text{O}_{24}(\text{OH})_2$	solid state rxn. of BaO , PbO , $\text{SrO} + \text{P}_2\text{O}_5$ in air at elevated temp.	x-ray diffractometry; infrared, broad-line NMR spectrometry; gravimetric anal.	illustrate nature of non-stoichiometric phases	T. Negas and P. Roth, "High Temperature Dehydroxylation of Apatitic Phosphates", J. Res. NBS, 72A, 783 (1968).
H_4PI PH_4I	$\text{PH}_3 + \text{HI} \rightarrow$	infrared, NMR (^1H , ^31P) spectrometry, vapor pressure	phase transitions and molecular motions in solids; toxic, moisture sensitive	T. C. Farrar and J. J. Rush, "Nuclear Magnetic Resonance and Neutron-Scattering Studies of Molecular Motions in Phosphonium Iodide", Proc. Second IMR Symp. (Molecular Dynamics and Structure of Solids), NBS Special Publ. 301 (1969), p. 245.
H_4PX PH_4Br PH_4Cl PH_4I	$\text{X} = \text{Br}, \text{Cl}, \text{I};$ volatile solid	NMR (^1H , ^31P), laser Raman spectrometry; vapor pressure, neutron inelastic scattering	model compounds, phase transitions and motions in solids	T. Teang, T. C. Farrar, and J. J. Rush, "Proton Magnetic Resonance and Hindered Rotation in Phosphonium Halides and Ammonium Iodide", J. Chem. Phys., 49, 4403 (1968).
				J. J. Rush, A. J. Melveger, T. C. Farrar and T. Tsang, "Laser-Raman Spectra and Hindered Rotation in the Phosphonium Halides", Chem. Phys. Letters, 2, 621 (1968).

H ₃ K ₂ Re K ₂ ReH ₉	supplied by A. P. Ginsberg, Bell Tele- phone Labs., Murray Hill, N. J.	neutron, x-ray diffractometry, NMR (¹ H) spectro- metry; elemental anal.	study of molecular dynamics of two crystal- lographically different anions; illustrates different reorientation energies	T. C. Farrar R. B. Johannesen	T. C. Farrar, T. Johannesen, "Internal Reorientation in K ₂ ReH ₉ via Wide- Line and Pulsed Proton Magnetic Resonance Studies", J. Chem. Phys., 51, 3595 (1969).
IrO ₂	oxidation of Ir by heat treat- ment in air and/or O ₂	x-ray diffracto- metry, phase equilibrium	illustrates behavior of container materials; dissociates at 1020°C in air to Ir+O ₂ ; see also O ₂ Ru	C. L. McDaniel S. J. Schneider	C. L. McDaniel and S. J. Schneider, "Phase Relations of the Ru-Ir-O ₂ System", J. Res. NBS, 72A, 213 (1968).
KNaO ₄ S KNaSO ₄	melt equimolar K ₂ SO ₄ +Na ₂ SO ₄ ; annealed over- night at 600°C	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 50.
K ₂ Mg ₂ O ₁₂ S ₃ K ₂ Mg ₂ (SO ₄) ₃	melt K ₂ SO ₄ + MgSO ₄ at 1000°C	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 40.
K ₂ Mn ₂ O ₁₂ S ₃ K ₂ Mn ₂ (SO ₄) ₃	melt K ₂ SO ₄ + MgSO ₄ ; annealed at 500°C for 15 hr.	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 43.
K ₂ Na ₄ O ₁₂ S ₃ K _{.67} Na _{1.33} (SO ₄) ₃	melt stoi- chiometric amts. K ₂ SO ₄ + Na ₂ SO ₄	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 48.

$K_2Ni_2O_7 \cdot 5H_2O$ $K_2Ni_2O_7 \cdot 5H_2O$	$K_2SO_4 + NiSO_4$ heated at 750°C, cooled slowly, ground, annealed at 550°C for 30 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 46.
$K_2O_4S_3Zn_2$ $K_2Zn_2(SO_4)_3$	$K_2SO_4 + ZnSO_4$ melted, ground, remelted	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 54.
$K_3NaO_8S_2$ $K_3Na(SO_4)_2$	stoichiometric K_2SO_4 + Na_2SO_4 melted, annealed at 700°C for 72 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 52.
$LiNaO_4S$ $LiNaSO_4$	equimolar Li_2SO_4 + Na_2SO_4 melted, annealed at 500°C overnight	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 24.
$LiNbO_3$	obtained from CIBA, Rare Metals Div., Summit, N.J.; recrystallized by W. S. Brower, pulled from melt, annealed under O_2 at 1100°C for 10 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 22.
Li_2O_4S Li_2SO_4	$Li_2SO_4 \cdot H_2O$ heated at 600°C for 24 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 26.

LuO ₄ V LuVO ₄	soln. Na ₃ VO ₄ + Lu ₂ (SO ₄) ₃ + ppt.; annealed at 1300°C for 15 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 37.
Na ₂ NiO ₈ S ₂ ·4H ₂ O Na ₂ Ni(SO ₄) ₂ ·4H ₂ O	crystallized from aq. soln. of Na ₂ SO ₄ + NiSO ₄ at room temp.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 68.
Na ₂ O ₈ S ₂ Zn·4H ₂ O Na ₂ Zn(SO ₄) ₂ ·4H ₂ O	crystallized from soln. of Na ₂ SO ₄ + ZnSO ₄ at room temp.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 72.
NbO ₄ Sb SbNbO ₄	solid state rxn. of Sb ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tube at 1000-1100°C	see BiNbO ₄	R. S. Roth J. L. Waring	See ref. under BiNbO ₄ .
"NbO ₃ P" "NbPO ₅ " 22Nb ₂ O ₅ ·P ₂ O ₅	solid state rxn. of Nb ₂ O ₅ +P ₂ O ₅ in sealed Pt. tube	x-ray powder diffractometry	E. M. Levin	E. M. Levin and R. S. Roth. "The System Niobium Pentoxide-Phosphorus Pentoxide", J. Solid State Chemistry <u>2</u> , (1970).
Nb ₂₄ O ₆₂ Ti TiNb ₂₄ O ₆₂	solid state rxn. of TiO ₂ + Nb ₂ O ₅ at 1450°C	x-ray powder and single crystal diffractometry; crystal structure detm.	R. S. Roth	R. S. Roth and A. D. Wadsley. "Mixed Oxides of Titanium and Niobium: The Crystal Structure of TiNb ₂₄ O ₆₂ (TiO ₂ ·12Nb ₂ O ₅)", Acta Cryst., <u>18</u> , 724 (1965).

RuO_4	powder	oxidation of Ru by heat treatment in air	x-ray diffractometry, phase equilibrium	illustrates behavior of container materials, dissociates at 1045°C in air to Ru + O_2 ; see also IrO_2	C. L. McDaniel S. J. Schneider	See ref. under IrO_4 .
$(1-x)\text{O}_2\text{Th} + x\text{CaO}$ $0 < x < 0.10$	porous bar	isostatically cold pressed and sintered 1 hr. at 1800°C	spectroscopic analysis	character of point defect; electrical and mechanical relaxation	J. Wachtman, Jr.	J. B. Wachtman, Jr., "Mechanical and Electrical Relaxation in ThO_2 Containing CaO ", Phys. Rev., <u>131</u> , 517 (1963).
$(1-x)\text{O}_2\text{Zr} + x\text{CaO}$ $0.04 < x < 0.20$	porous bar	isostatically cold pressed and sintered 1 hr. at 1800°C	porosity, grain size	spans range of stability of cubic solid solution and includes 2-phase region below $x=0.10$ as test of change in character of point defects	J. Wachtman, Jr.	J. B. Wachtman, Jr. and W. C. Corwin, "Internal Friction in ZrO_2 Containing CaO ", J. Res. NBS, <u>69A</u> , 457 (1965).
O_4PrV PrVO_4		$\text{Pr}_2(\text{C}_2\text{O}_4)_3 + \text{V}_2\text{O}_5$ heated at 850°C for 30 min.	x-ray diffractometry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 40.
O_4SbTa SbTaO_4		solid state rxn. of $\text{Sb}_2\text{O}_3 + \text{Ta}_2\text{O}_5$ in sealed Pt tube at 1000-1200°C	see BiNbO_4		R. S. Roth J. L. Waring	See ref. under BiNbO_4 .
O_4SbV SbVO_4		solid state rxn. of $\text{Sb}_2\text{O}_3 + \text{V}_2\text{O}_5$ in sealed Pt tube at 700-855°C	see BiNbO_4		R. S. Roth J. L. Waring	See ref. under BiNbO_4 .

O_4SmV $SmVO_4$	$Sm_2(C_2O_4)_3 + V_2O_5$ heated at $850^\circ C$ for 45 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 47.
O_4TbV $TbVO_4$	$Tb_2(C_2O_4)_3 + V_2O_5$ heated at $1400^\circ C$ for 1 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 56.
O_4TmV $TmVO_4$	$Tm_2(C_2O_4)_3 + V_2O_5$ heated at $1400^\circ C$ for 1 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 57.
O_4YbV $YbVO_4$	$Yb_2(C_2O_4)_3 + V_2O_5$ heated at $1400^\circ C$ for 1 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 58.
Pt	high-purity rod, $1/8$ in. dia. x 1.5 in.	Langmuir detm. of vapor pressure as function of temp.; detm. of heat of sublimation	R. C. Paule	See ref. under Ag.
	special prepn. for Office of Standard Reference Materials, proposed SRM # 747			
	SRM # 747 SRM # 680	vapor pressure Langmuir method	E. R. Plante	See above ref.
Re	rod, .25 cm. dia. x 1.5 cm.	vapor pressure	E. R. Plante	E. R. Plante and R. Szwarc, "Vapor Pressure and Heat of Sublimation of Rhenium", J. Res. NBS, 70A, 175 (1966).

$V_2O_5 \cdot 4Y_2O_3$
 $4Y_2O_3 \cdot V_2O_5$
 $V_2O_5 \cdot 5Y_2O_3$
 $5Y_2O_3 \cdot V_2O_5$

$\sim 50\%$

solid state
 rxn. in
 sealed Pt
 tube

x-ray powder
 diffractometry

phosphors

E. M. Levin
 E. M. Levin
 E. M. Levin, "System
 Y_2O_3 - V_2O_5 ", J. Am.
 Ceram. Soc., 50, 381
 (1967).

W

high-
 purity
 rod, 1/4
 in. dia. x
 2-1/2 in.

polycry-
 stalline
 rod

special
 Depn. for
 Office of
 Standard
 Reference
 Materials,
 proposed
 SRM # 749

commercial

see Pt

vapor pressure,
 Langmuir
 method

vapor
 pressure
 standard;
 container for
 high-temp.
 studies

R. C. Paule

E. R. Plante

See ref. under Pt.

R. Swarc, E. R.
 Plante, and J. J.
 Diamond, "Vapor Pres-
 sure and Heat of
 Sublimation of
 Tungsten", J. Res.
 NBS, 69A, 417
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NBS STAFF MEMBERS
TO BE CONTACTED
FOR ADDITIONAL
INFORMATION

REFERENCES (Author,
Title, Journal)

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	REFERENCES (Author, Title, Journal)
$Al_2O_3-WO_3$ entire system	polycrystal- line, 1-25 μ	solid state rxn. of com- ponents in sealed Pt tubes	Phase equilibria; x-ray powder and single crystal diffraction studies	phase equilibria	J. L. Waring, "Phase Equilibria in the System Aluminum Oxide- Tungsten Oxide", J. Am. Ceram. Soc., 48, 493 (1965). D. C. Craig and N. C. Stephenson, "A Structural Study in the System Al_2O_3 - WO_3 ", Acta Cryst., B24, 1250 (1969).
$As_2O_5-9Nb_2O_5$ partial system	polycrystal- line, 1-25 μ	solid state rxn. in sealed Pt tube	phase equilibria; x-ray diffraction powder and single crystal structure	illustrates nature of apparent iso- structural phases in related systems; see also: $As_2O_5-9Ta_2O_5$, $GeO_2-9Nb_2O_5$, $9Nb_2O_5-P_2O_5$, $9Nb_2O_5-V_2O_5$, $2Nb_2O_5-Ta_2O_5$, $2Nb_2O_5-9Ta_2O_5$, $9Ta_2O_5-V_2O_5$	J. L. Waring and R. S. Roth, "Tetragonal Phases of the General Type $10M_2O_5 \cdot 90M_2O_5$ apparently isostruct- tural with Ta_2O_5 . Nb_2O_5 ", Acta Cryst., 17, 455 (1964). R. S. Roth and A. D. Wadsley, "The Crystal Structure of $PbNb_9O_{25}$ ($P_2O_5 \cdot 9Nb_2O_5$)", Acta Cryst., 18, 643 (1965).
$As_2O_5-9Ta_2O_5$	partial system, see comments and ref. under $As_2O_5-9Nb_2O_5$				
B_2O_3 MB03	solid solns. of rare-earth borates, M = Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Sm, Tm, Y, Yb; polycrystal- line	solid state rxns. of mix- tures of M_2O_3 + B_2O_3 in sealed Pt tubes	x-ray powder diffractometry; high-temp. x-ray phase equilibria	most specimens exhibit polymorphic inversions	R. S. Roth J. L. Waring E. M. Levin R. S. Roth, J. L. Waring, and E. M. Levin, "Polymorphism of ABO_3 -Type Rare Earth Borate Solid Solutions", Rare Earth Research II, Gordon & Breach, N.Y., (1964), P. 153.

BaFeO _{3-n} BaO. "iron oxide"	0.4x10 ⁻⁵ ; system in the vicinity of 1:1 cation ratio; poly- crystalline, 1-25 μ	solid state rxn. in Au tubes under O ₂	phase equilibria; x-ray diffractometry	illustrated influence of stoichiometry on structure	T. Negas ² R. S. Roth	T. Negas and R. S. Roth, "Synthesis of Barium Ferrates in Oxygen", J. Res. NBS, 73A, (1969).
BaO-Nb ₂ O ₅ entire system	polycrystal- line	solid state rxns. of com- ponents in sealed Pt tubes	x-ray powder diffractometry; phase equilibria		R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Phase Equi- librium Relations in the Binary System Barium Oxide-Niobium Pentoxide", J. Res. NBS, 65A, 337 (1961).
BaO-Pt entire system	powders	solid state rxn. conducted in air in Pt and Au con- tainers	x-ray diffracto- metry, phase equilibria	all inter- mediate phases dis- sociate; illustrates behavior of container material	S. J. Schneider C. L. McDaniel	S. J. Schneider and C. L. McDaniel, "The BaO-Pt System in Air", J. Am. Ceram. Soc., 52, 518 (1969).
BaSi ₂ O ₅ -Ba ₂ Si ₃ O ₈ entire system	polycrystal- line	solid state rxn.	x-ray powder diffractometry; phase equilibria; single crystal x-ray diffractometry		R. S. Roth E. M. Levin	R. S. Roth and E. M. Levin, "Phase Equi- libria in the System Barium Disilicate- Dibarium Trisilicate", J. Res. NBS, 52, 193 (1959).
Bi ₂ O ₃ -Nb ₂ O ₅ entire system	polycrystal- line	solid state rxns. in sealed Pt tubes	x-ray powder diffractometry; phase equilibria		R. S. Roth J. L. Waring	R. S. Roth and E. M. Levin, "Polymorphism in Barium Disilicate", Am. Mineral., 44, 452 (1959).
						R. S. Roth and J. L. Waring, "Phase Equi- librium Relations in the Binary System Bismuth Sesquioxide- Niobium Pentoxide", J. Res. NBS, 66A, 451 (1962).

$\text{Cr}_2\text{O}_3\text{-IrO}_2$ entire system	powders	solid state rxns. in Pt, Vycor, Ir containers, conducted in air	x-ray diffractometry; phase equilibria	illustrates behavior of container materials	C. L. McDaniel S. J. Schneider "Phase Relations between Cr_2O_3 and IrO_2 in Air", J. Am. Ceram. Soc., 49, 285 (1966).
" CrO_n "-SrO "chromium oxide" - SrO entire system	polycrystalline, 1-25 μ	solid state rxns. in air and O_2	x-ray diffractometry; gravimetric anal.; phase equilibria	illustrates redox behavior of Cr in system	T. Negas and R. S. Roth, "The System SrO -"chromium oxide" in Air and Oxygen", J. Res. NBS, 73A (1969).
$\text{Dy}_2\text{O}_3\text{-TiO}_2$	partial system, see comments and ref.	under $\text{Gd}_2\text{O}_3\text{-TiO}_2$			
$\text{Er}_2\text{O}_3\text{-2TiO}_2$	partial system, see comments and ref.	under $\text{Gd}_2\text{O}_3\text{-TiO}_2$			
$\text{Eu}_2\text{O}_3\text{-TiO}_2$ $\text{Eu}_2\text{O}_3\text{-2TiO}_2$	partial system, see comments and ref.	under $\text{Gd}_2\text{O}_3\text{-TiO}_2$			
$\text{Gd}_2\text{O}_3\text{-TiO}_2$ entire system	polycrystalline, 1-25 μ	solid state rxn. in unsealed Pt tubes	x-ray diffractometry; polarizing microscopy; phase equilibria	illustrates nature of phases form and relationship to analogous systems; see also: $\text{Dy}_2\text{O}_3\text{-TiO}_2$, $\text{Er}_2\text{O}_3\text{-2TiO}_2$, $\text{Eu}_2\text{O}_3\text{-TiO}_2$, $\text{Eu}_2\text{O}_3\text{-2TiO}_2$, $\text{Ho}_2\text{O}_3\text{-2TiO}_2$, $\text{Lu}_2\text{O}_3\text{-2TiO}_2$, $\text{Sm}_2\text{O}_3\text{-TiO}_2$, $2\text{TiO}_2\text{-Tm}_2\text{O}_3$	J. L. Waring and S. J. Schneider, "Phase Equilibrium Relationships in the System $\text{Gd}_2\text{O}_3\text{-TiO}_2$ ", J. Res. NBS, 69A, 255 (1965).
$\text{GeO}_2\text{-9Nb}_2\text{O}_5$	partial system, see comments and refs.	under $\text{As}_2\text{O}_5\text{-9Nb}_2\text{O}_5$; also see $\text{GeO}_2\text{-9Nb}_2\text{O}_5$ in SECTION V			
$\text{Ho}_2\text{O}_3\text{-2TiO}_2$	partial system, see comments and ref.	under $\text{Gd}_2\text{O}_3\text{-TiO}_2$			

<p>$\text{IrO}_2\text{-SnO}_2$</p> <p>entire system</p> <p>$\text{IrO}_2\text{-TiO}_2$</p> <p>entire system</p>	<p>powders</p> <p>solid state rxns. in Pt, Vycor, and Ir containers, conducted in air</p>	<p>x-ray diffractometry, phase equilibria</p>	<p>illustrates behavior of container materials</p>	<p>C. L. McDaniel S. J. Schneider</p> <p>"Phase Relations in the Systems $\text{TiO}_2\text{-IrO}_2$ and $\text{SnO}_2\text{-IrO}_2$ in Air", J. Res. NBS, <u>71A</u>, 119 (1967).</p>	<p>C. L. McDaniel and S. J. Schneider, "Phase Relations in the Systems $\text{TiO}_2\text{-IrO}_2$ and $\text{SnO}_2\text{-IrO}_2$ in Air", J. Res. NBS, <u>71A</u>, 119 (1967).</p>
<p>$\text{Lu}_2\text{O}_3\text{-PdO}$</p> <p>entire system</p>	<p>powders</p> <p>solid state rxns. in Pt, Vycor containers</p>	<p>x-ray diffractometry, phase equilibria</p>	<p>all intermediate phases dissociate; illustrates behavior of container materials</p>	<p>C. L. McDaniel S. J. Schneider</p> <p>"Phase Relations between Palladium Oxide and the Rare-Earth Sesquioxides in Air", J. Res. NBS, <u>72A</u>, 27 (1968).</p>	<p>C. L. McDaniel and S. J. Schneider, "Phase Relations between Palladium Oxide and the Rare-Earth Sesquioxides in Air", J. Res. NBS, <u>72A</u>, 27 (1968).</p>
<p>$\text{Lu}_2\text{O}_3\text{-2TiO}_2$</p>	<p>partial system, see comments and ref. under $\text{Gd}_2\text{O}_3\text{-TiO}_2$</p>				
<p>$9\text{Nb}_2\text{O}_5\text{-P}_2\text{O}_5$</p>	<p>partial system, see comments and refs. under $\text{As}_2\text{O}_5\text{-9Nb}_2\text{O}_5$; also see "Nb$_2\text{O}_5$P" in SECTION V</p>				
<p>$2\text{Nb}_2\text{O}_5\text{-Ta}_2\text{O}_5$</p>	<p>partial system, see comments and refs. under $\text{As}_2\text{O}_5\text{-9Nb}_2\text{O}_5$</p>				
<p>$\text{Nb}_2\text{O}_5\text{-V}_2\text{O}_5$</p> <p>entire system</p>	<p>polycrystalline, 1-25 μ sealed Pt tubes</p> <p>solid state rxns. in sealed Pt tubes</p>	<p>x-ray powder diffractometry; phase equilibria</p>	<p>illustrates nature of multi-phase formation and non-stoichiometric phases; see also $9\text{Nb}_2\text{O}_5\text{-V}_2\text{O}_5$</p>	<p>J. L. Waring R. S. Roth</p> <p>J. L. Waring and R. S. Roth, "Phase Equilibria in the System Vanadium Oxide-Niobium Oxide", J. Res. NBS, <u>69A</u>, 119 (1964).</p>	<p>J. L. Waring and R. S. Roth, "Phase Equilibria in the System Vanadium Oxide-Niobium Oxide", J. Res. NBS, <u>69A</u>, 119 (1964).</p>
<p>$9\text{Nb}_2\text{O}_5\text{-V}_2\text{O}_5$</p>	<p>partial system, see comments and refs. under $\text{As}_2\text{O}_5\text{-9Nb}_2\text{O}_5$; also see $\text{Nb}_2\text{O}_5\text{-V}_2\text{O}_5$</p>				
<p>$\text{P}_2\text{O}_5\text{-9Ta}_2\text{O}_5$</p>	<p>partial system, see comments and refs. under $\text{As}_2\text{O}_5\text{-9Nb}_2\text{O}_5$</p>				
<p>$\text{Sm}_2\text{O}_3\text{-TiO}_2$</p>	<p>partial system, see comments and ref. under $\text{Gd}_2\text{O}_3\text{-TiO}_2$</p>				

J. L. Waring and
R. S. Roth, "Effects
of Oxide Additions on
the Polymorphism of
Tantalum Pentoxide
(System $Ta_2O_5-TiO_2$),"
J. Res. NBS, 71A, 175
(1967).

J. L. Waring
R. S. Roth
illustrates
various stable
and metastable
phases formed
in the system

x-ray powder
diffractometry;
high-temp. x-ray
powder diffracto-
metry; phase
equilibria

solid state
rxns. in
sealed Pt
tubes

polycrystal-
line

$Ta_2O_5-TiO_2$
entire system

$3Ta_2O_5-V_2O_5$ partial system, see comments and refs. under $As_2O_5-9Nb_2O_5$

$2TiO_2-Tm_2O_3$ partial system, see comments and ref. under $Gd_2O_3-TiO_2$

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